

# **Recommended Practice Standard Procedure for Field Testing Water-Based Drilling Fluids**

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**Note:**

This first edition supersedes the Twelfth Edition of RP13B, dated September 1, 1988. This edition includes revisions adopted at the 1988 and 1989 API Standardization Conferences and subsequently approved by letter ballot. With this first edition, RP13B has been divided into two separate documents, one for water-based muds and another for oil-based muds.

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## RECOMMENDED PRACTICE STANDARD PROCEDURE FOR FIELD TESTING WATER-BASED DRILLING FLUIDS

### Foreword

- a. This recommended practice is under the jurisdiction of the API Committee on Standardization of Drilling Fluid Materials.
- b. The purpose of this recommended practice is to provide standard procedures for the testing of water-based drilling fluids. It is not a detailed manual on mud control procedures. It should be remembered that the agitation history and temperature of testing have a profound effect on mud properties.
- c. This recommended practice is organized to follow the tests as listed on the API Drilling Mud Report form (API RP 13G, Second Edition, May 1982). Additional tests are given in the Appendix of this recommended practice.
- d. Metric "SI" unit equivalents have been included in this publication in parentheses following the U.S. customary units.
- e. Additional publications under jurisdiction of this committee: Spec 13A, Specification for Drilling Fluid Materials, covers specifications and test procedures for barite, hematite, bentonite, nontreated bentonite, attapulgitite, and sepiolite, starch, technical-grade low viscosity CMC, technical-grade high viscosity CMC, and OCMA grade bentonite.

RP 13B-2 Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluids

Bul 13C Bulletin on Drilling Fluids Processing Equipment

Bul 13D Bulletin on the Rheology of Oil Well Drilling Fluids

RP 13E Recommended Practice for Shale Shaker Screen Cloth Designation

RP 13G Recommended Practice for Drilling Mud Report Form

RP 13I Recommended Practice for Laboratory Testing of Drilling Fluids

RP 13J Recommended Practice for Testing Heavy Brines

RP 13K Recommended Practice for Chemical Analysis of Barite

## SECTION 1 MUD WEIGHT (DENSITY)

**1.1 Description.** This test procedure is a method for determining the weight of a given volume of liquid. Mud weight may be expressed as pounds per gallon (lb/gal), pounds per cubic foot (lb/ft<sup>3</sup>), grams per cubic centimeter (g/cm<sup>3</sup>), or kilograms per cubic meter (kg/m<sup>3</sup>).

### 1.2 Equipment

a. Any instrument of sufficient accuracy to permit measurement within  $\pm 0.1$  lb/gal (or 0.5 lb/ft<sup>3</sup>, 0.01 g/cm<sup>3</sup>, 10 kg/m<sup>3</sup>) may be used. The mud balance (Fig. 1.1 and 1.2) is the instrument generally used for mud weight determinations. The mud balance is designed such that the mud cup, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (Attachments for extending the range of the balance may be used when necessary).

b. Thermometer: 32-220°F (0-105°C)

### 1.3 Procedure

a. The instrument base should be set on a flat, level surface.

b. Measure the temperature of the mud and record on the Drilling Mud Report form.

c. Fill the clean, dry cup with mud to be tested; put the cap on the filled mud cup and rotate the cap until it is firmly seated. Insure that some of the mud is expelled through the hole in the cap in order to free any trapped air or gas (see Appendix D for Air Removal).

d. Holding cap firmly on mud cup (with cap hole covered), wash or wipe the outside of the cup clean and dry.

e. Place the beam on the base support and balance it by moving the rider along the graduated scale. Balance is achieved when the bubble is under the center line.

f. Read the mud weight at edge of the rider toward the mud cup. Make appropriate corrections when a range extender is used.

**1.4 Procedure-Calibration.** The instrument should be calibrated frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/ft<sup>3</sup> (1000 kg/m<sup>3</sup>) at 70°F (21°C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

### 1.5 Calculation

a. Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft<sup>3</sup> (0.01 g/cm<sup>3</sup>, 10 kg/m<sup>3</sup>).

b. To convert the reading to other units, use the following:

$$\text{Density} = \text{g/cm}^3 = \frac{\text{lb/ft}^3}{62.3} = \frac{\text{lb/gal}}{8.345} \quad (\text{a})$$

$$\text{kg/m}^3 = (\text{lb/ft}^3) (16) = (\text{lb/gal}) (120) \quad (\text{b})$$

$$\text{Mud gradient} = \frac{\text{lb/ft}^3}{144} \quad \frac{\text{lb/gal}}{19.24} \quad \text{or} \quad \frac{\text{kg/m}^3}{2309} \quad (\text{c})$$

TABLE 1.1  
DENSITY CONVERSION

1	2	3	4
pounds per gallon (lb/gal)	pounds per cubic foot (lb/ft <sup>3</sup> )	grams per cubic centimeter (g/cm <sup>3</sup> )*	kilograms per cubic meter (kg/m <sup>3</sup> )
6.5	48.6	0.78	780
7.0	52.4	0.84	840
7.5	56.1	0.90	900
8.0	59.8	0.96	960
8.3	62.3	1.00	1000
8.5	63.6	1.02	1020
9.0	67.3	1.08	1080
9.5	71.1	1.14	1140
10.0	74.8	1.20	1200
10.5	78.5	1.26	1260
11.0	82.3	1.32	1320
11.5	86.0	1.38	1380
12.0	89.8	1.44	1440
12.5	93.5	1.50	1500
13.0	97.2	1.56	1560
13.5	101.0	1.62	1620
14.0	104.7	1.68	1680
14.5	108.5	1.74	1740
15.0	112.5	1.80	1800
15.5	115.9	1.86	1860
16.0	119.7	1.92	1920
16.5	123.4	1.98	1980
17.0	127.2	2.04	2040
17.5	130.9	2.10	2100
18.0	134.6	2.16	2160
18.5	138.4	2.22	2220
19.0	142.1	2.28	2280
19.5	145.9	2.34	2340
20.0	149.6	2.40	2400
20.5	153.3	2.46	2460
21.0	157.1	2.52	2520
21.5	160.8	2.58	2580
22.0	164.6	2.64	2640
22.5	168.3	2.70	2700
23.0	172.1	2.76	2760
23.5	175.8	2.82	2820
24.0	179.5	2.88	2880

\*Same as specific gravity (sg).

## ALTERNATE MUD WEIGHT METHOD

1.6 Description. The density of a mud containing entrained air or gas can be determined more accurately by using the pressurized fluid density balance as described in this section. The pressurized fluid density balance is similar in operation to the conventional mud balance, the difference being that the slurry sample can be placed in a fixed volume sample cut under pressure.

The purpose of placing the sample under pressure is to minimize the effect of entrained air or gas upon slurry density measurements. By pressurizing the sample cup, any entrained air or gas will be decreased to a negligible volume, thus providing a slurry density measurement more closely in agreement with that which will be realized under downhole conditions.

### 1.7 Equipment

a. Any instrument of sufficient accuracy to permit measurement within  $\pm 0.1$  lb/gal (or  $0.5$  lb/ft<sup>3</sup>,  $0.01$  g/cm<sup>3</sup>,  $10$  kg/m<sup>3</sup>) may be used. The pressurized mud balance (Fig. 1.3 and 1.4) is the instrument generally used for pressurized balance mud weight determinations. The pressurized mud balance is designed such that the mud cup and screw on lid, at one end of the beam, is balanced by a fixed counterweight at the other end, with a sliding-weight rider free to move along a graduated scale. A level-bubble is mounted on the beam to allow for accurate balancing. (See Figs. 1.3 and 1.4)

b. Thermometer: 32-220°F (0-105°C)

### 1.8 Procedure

a. Fill the sample cup to a level slightly below the upper edge of the cup [approximately  $\frac{1}{4}$  inch (6.4 mm)].

b. Place the lid on the cup with the attached check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry will be expelled through the check valve. When the lid has been placed on the cup, pull the check valve up in the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.

c. The pressurizing plunger is similar in operation to a syringe. Fill the plunger by submersing its end in the slurry with the piston rod in the completely inward position. The piston rod is then drawn upward thereby filling the cylinder with slurry. This volume should be expelled with the plunger action and refilled with fresh slurry sample to ensure that this plunger volume is not diluted with liquid remaining from the last clean up of the plunger mechanism.

d. Push the nose of the plunger onto the mating O-ring surface of the cap valve. Pressurize the sample cup

by maintaining a downward force on the cylinder housing in order to hold the check valve down (open) and at the same time forcing the piston rod inward. Approximately 50 pounds (225 N) force or greater should be maintained on the piston rod. Figure 1.4.

e. The check valve in the lid is pressure actuated; when pressure is placed within the cup, the check valve is pushed upward into the closed position. To close the valve gradually ease up on the cylinder housing while maintaining pressure on the piston rod. When the check valve closes, release pressure on the piston rod before disconnecting the plunger.

f. The pressurized slurry sample is now ready for weighing. Rinse the exterior of the cup and wipe dry. Place instrument on the knife edge as illustrated. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the attached bubble is centered between the two black marks. Read the density from one of the four calibrated scales on the arrow side of the sliding weight. The density can be read directly in units of lb/gal, specific gravity, psi/1000 ft and lb/ft<sup>3</sup>.

g. To release the pressure inside the cup reconnect the empty plunger assembly and push downward on the cylinder housing.

h. Clean the cup and rinse thoroughly with water. For best operation in water-based slurries, the valve should be greased frequently with waterproof grease.

### 1.9 Procedure-Calibration

a. Calibrate the instrument frequently with fresh water. Fresh water should give a reading of 8.3 lb/gal or 62.3 lb/cu ft<sup>3</sup> (1000 kg/m<sup>3</sup>) at 70°F (21°C). If it does not, adjust the balancing screw or the amount of lead shot in the well at the end of the graduated arm as required.

### 1.10 Calculation

a. Report the mud weight to the nearest 0.1 lb/gal or 0.5 lb/ft<sup>3</sup> (0.01 gm/cm<sup>3</sup>, 10 kg/m<sup>3</sup>).

b. To convert the reading to other units, use the following:

$$\text{Density} = \text{g/cm}^3 = \frac{\text{lb/ft}^3}{62.43} = \frac{\text{lb/gal}}{8.345} \quad (a)$$

$$\text{kg/m}^3 = (\text{lb/ft}^3) (16) = (\text{lb/gal}) (120) \quad (b)$$

$$\text{Mud gradient,} = \frac{\text{lb/ft}^3}{144} = \frac{\text{lb/gal}}{19.24} = \frac{\text{kg/m}^3}{2309} \quad (c)$$

c. A list of Density Conversions may be found in Table 1.1.

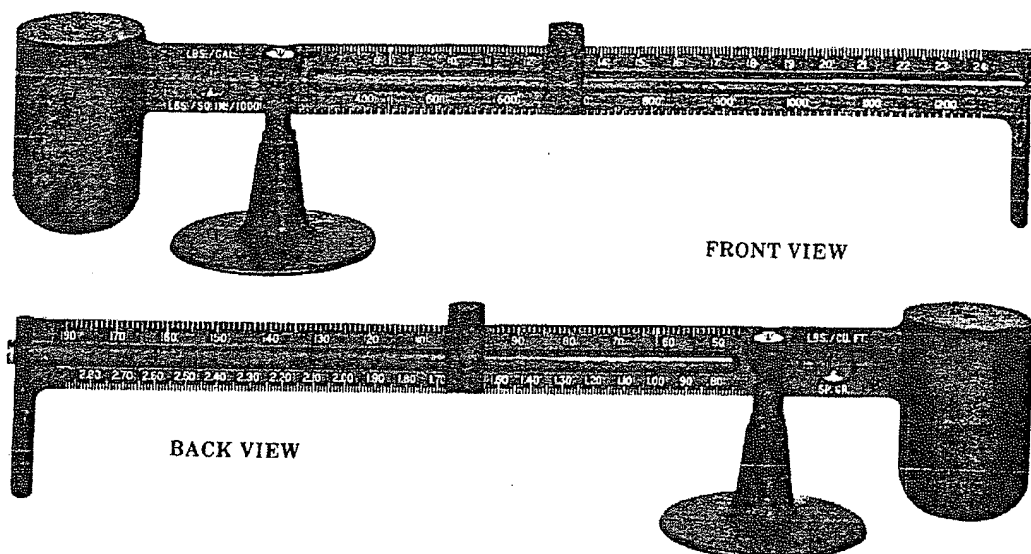


FIG. 1.1  
MUD BALANCE

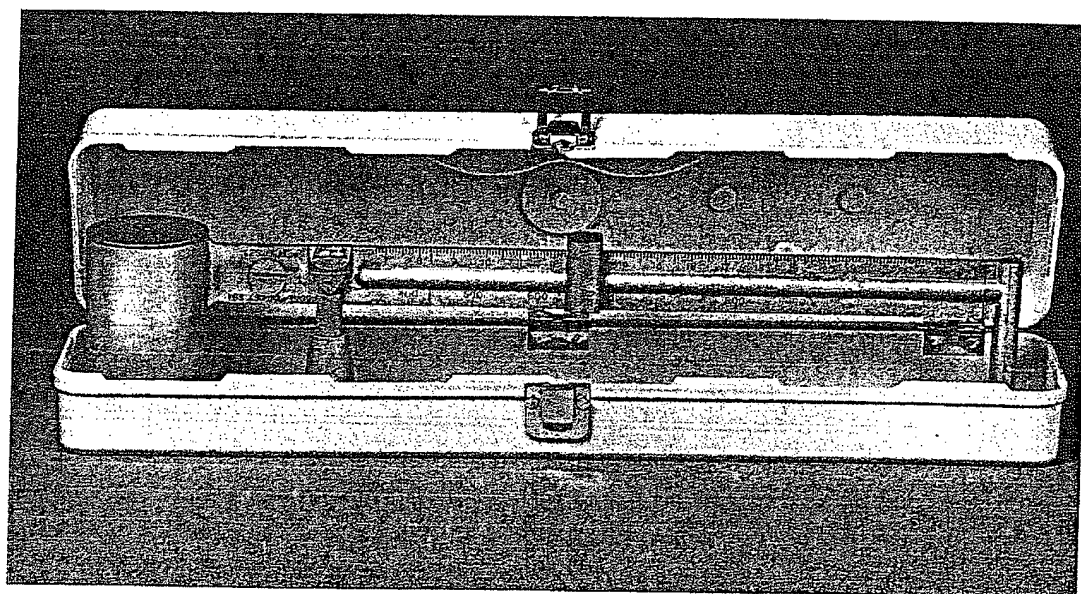


FIG. 1.2  
MUD BALANCE AND CASE



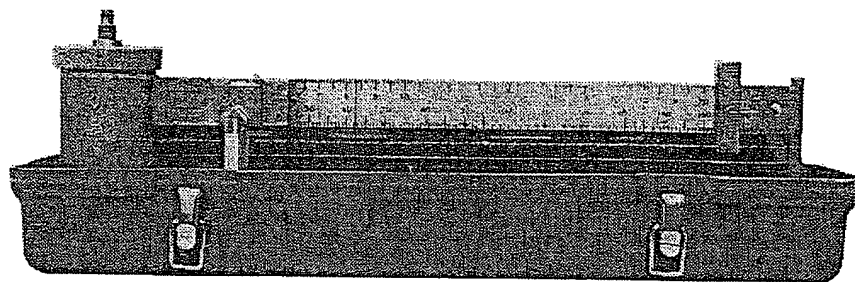


FIG 1.3  
PRESSURIZED FLUID DENSITY BALANCE

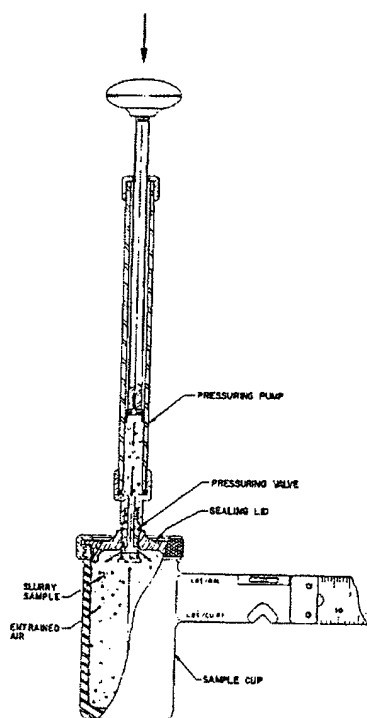


FIG 1.4  
TYPICAL PRESSURIZED FLUID DENSITY BALANCE DIAGRAM

## SECTION 2 VISCOSITY AND GEL STRENGTH

### 2.1 Description

a. The following instruments are used to measure viscosity and/or gel strength of drilling fluids:

1. Marsh Funnel — a simple device for indicating viscosity on a routine basis.
2. Direct-indicating viscometer — a mechanical device for measurement of viscosity at varying shear rates.

b. Viscosity and gel strength are measurements that relate to the flow properties of muds. The study of deformation and flow of matter is rheology. An in-depth discussion of rheology is made in API Bulletin 13D: *The Rheology of Oil-Well Drilling Fluids*.

### MARSH FUNNEL

#### 2.2 Equipment

##### a. Marsh Funnel

A Marsh Funnel (see Fig. 2.1) is calibrated to out-flow one quart (946 cm<sup>3</sup>) of fresh water at a temperature of 70 ± 5°F (21 ± 3°C) in 26 ± 0.5 seconds. A graduated cup is used as a receiver.

#### Specifications

##### Funnel Cone

Length	12.0 in. (305 mm)
Diameter	6.0 in. (152 mm)
Capacity to bottom of screen	1500 cm <sup>3</sup>

##### Orifice

Length	2.0 in. (50.8 mm)
Inside Diameter	3/16 in. (4.7 mm)
Screen	12 mesh
Has	1/16 in. (1.6 mm) openings and is fixed at a level 3/4 in. (19.0 mm) below top of funnel.

##### b. Graduated cup: one-quart

##### c. Stopwatch

##### d. Thermometer: 32-220°F (0-105°C)

### 2.3 Procedure

a. Cover the funnel orifice with a finger and pour freshly sampled drilling fluid through the screen into the clean, upright funnel. Fill until fluid reaches the bottom of the screen.

b. Remove finger and start stopwatch. Measure the time for mud to fill to one-quart (946 cm<sup>3</sup>) mark of the cup.

c. Measure temperature of fluid in degrees F (C).

d. Report the time to nearest second as Marsh Funnel viscosity. Report the temperature of fluid to nearest degree F (C).

### DIRECT-INDICATING VISCOMETER

#### 2.4 Equipment

a. Direct-indicating viscometers are rotational types of instruments powered by an electric motor or a hand crank. Drilling fluid is contained in the annular space between two concentric cylinders. The outer cylinder or rotor sleeve is driven at a constant rotational velocity (rpm). The rotation of the rotor sleeve in the fluid produces a torque on the inner cylinder or bob. A torsion spring restrains the movement of the bob, and a dial attached to the bob indicates displacement of the bob.

Instrument constants have been adjusted so that plastic viscosity and yield point are obtained by using readings from rotor sleeve speeds of 300 rpm and 600 rpm.

#### Specifications — Direct-indicating viscometer

##### Rotor Sleeve

Inside diameter	1.450 in. (36.83 mm)
Total length	3.425 in. (87.00 mm)
Scribed line	2.30 in. (58.4 mm)

above the bottom of sleeve.

Two rows of 1/8 in. (3.18 mm) holes spaced 120 deg (2.09 radians) apart, around rotor sleeve just below scribed line.

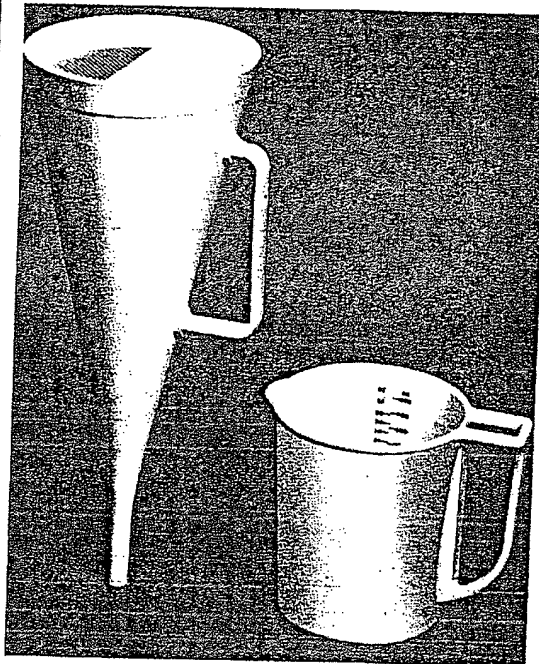


FIG. 2.1  
MARSH FUNNEL AND CUP

Bob	
Diameter.....	1.358 in. (34.49 mm)
Cylinder length.....	1.496 in. (38.00 mm)
Bob is closed with a flat base and a tapered top.	
Torsion spring constant	
386 dyne-cm/degree deflection	
Rotor speeds	
High speed.....	600 rpm
Low speed.....	300 rpm

The following are types of viscometers used to test drilling fluids:

1. Hand-cranked instrument (Fig. 2.2) has speeds of 300 rpm and 600 rpm. A knob on the hub of the speed-change lever is used to determine gel strength.
2. The 12-volt, motor driven instrument (Fig. 2.3) also has speeds of 300 and 600 rpm. A governor-release switch permits high shearing before measurement, and a knurled hand-wheel is used to determine gel strength.
3. The 115-volt instrument (Fig. 2.4) is powered by a two-speed synchronous motor to obtain speeds of 3, 6, 100, 200, 300 and 600 rpm. The 3-rpm speed is used to determine gel strength.
4. The variable speed 115-volt or 240-volt instrument (Fig. 2.5) is motor powered to obtain all speeds between 1 and 625 rpm. The 3-rpm speed is used to determine gel strength.

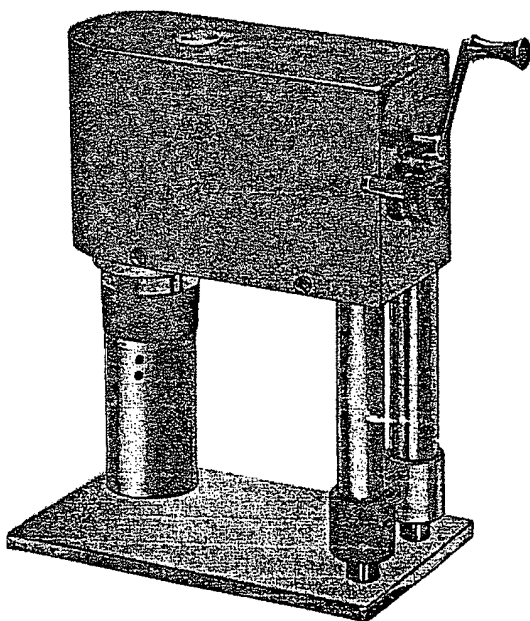


FIG. 2.2  
HAND-CRANK VISCOMETER

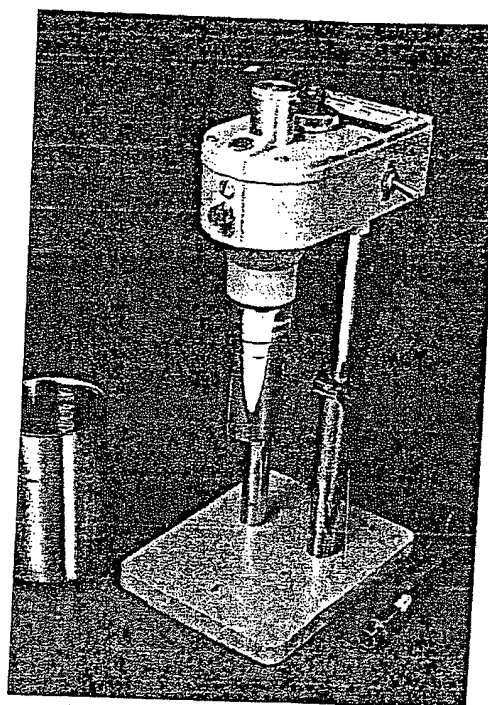


FIG. 2.3  
12-VOLT MOTOR-DRIVEN VISCOMETER

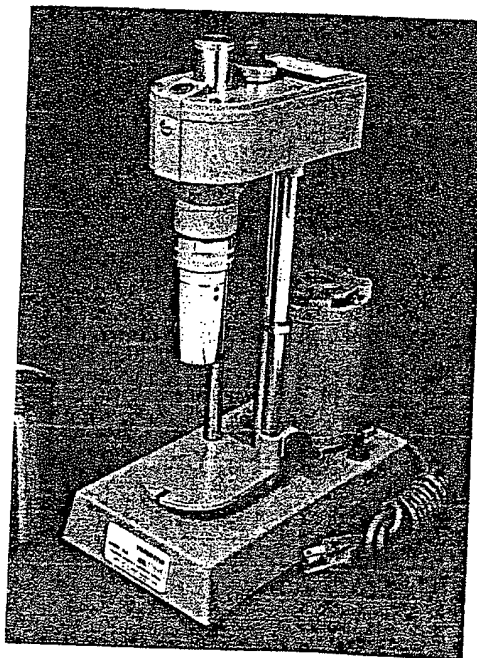


FIG. 2.4  
115-VOLT MOTOR-DRIVEN VISCOMETER

- b. Stopwatch
- c. Suitable container, e.g., the cup provided with the viscometer.
- d. Thermometer: 32-220°F (0-105°C)

### 2.5 Procedure

**CAUTION:** Maximum operating temperature is 200°F (93°C). If fluids above 200°F (93°C) are to be tested, a solid metal bob or a hollow metal bob, with a completely dry interior, should be used. Liquid trapped inside a hollow bob may vaporize when immersed in high temperature fluid and cause the bob to explode.

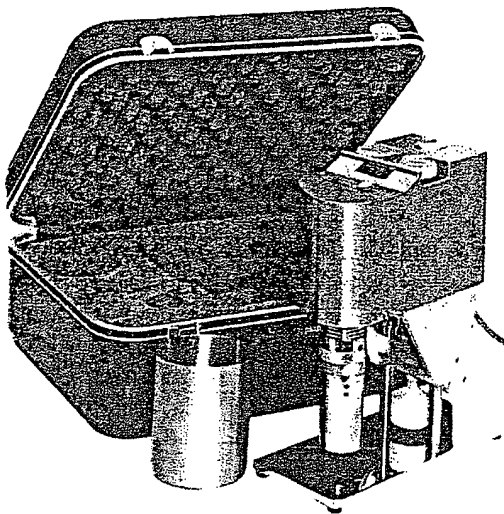


FIG. 2.5  
VARIABLE SPEED VISCOMETER

a. Place a sample in container and immerse the rotor sleeve exactly to the scribed line. Measurements in the field should be made with minimum delay (within five minutes, if possible) and at a temperature as near as practical to that of the mud at the place of sampling (not to differ more than 10°F (6°C)). The place of sampling should be stated on the report.

b. Record the temperature of the sample.

c. With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.

d. Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.

e. Stir drilling fluid sample for 10 seconds at high speed.

f. Allow drilling fluid sample to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after starting rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft<sup>2</sup> (Pa).

g. Restir the drilling fluid sample at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in Par. 2.5f and report the maximum reading as 10 minute gel in lb/100 ft<sup>2</sup> (Pa).

### 2.6 Calculation

$$\text{Plastic Viscosity, cP} = \left[ \begin{array}{c} 600 \text{ rpm} \\ \text{reading} \end{array} \right] - \left[ \begin{array}{c} 300 \text{ rpm} \\ \text{reading} \end{array} \right] \quad (a)$$

$$\text{Yield Point, lb/100 ft}^2 = \left[ \begin{array}{c} 300 \text{ rpm} \\ \text{reading} \end{array} \right] - \text{Plastic Viscosity} \quad (b)$$

$$\text{Apparent Viscosity, cP} = \frac{600 \text{ rpm reading}}{2} \quad (c)$$

## SECTION 3 FILTRATION

### 3.1 Description

a. Measurement of the filtration behavior and wall-cakebuilding characteristics of a mud are fundamental to drilling fluid control and treatment, as are the characteristics of the filtrate, such as oil, water, or emulsion content.

b. These characteristics are affected by the types and quantities of solids in the fluid and their physical and chemical interactions, which, in turn, are affected by temperature and pressure. Therefore, tests are run at both low-pressure/low temperature and high-pressure/high-temperature, and each requires different equipment and techniques.

### LOW-TEMPERATURE/LOW-PRESSURE TEST

#### 3.2 Equipment — Low-Temperature/Low-Pressure Test

a. A filter press consists mainly of a cylindrical mud cell having an inside diameter of 3 in. (76.2 mm) and a height of at least 2.5 in. (64.0 mm). This chamber is made of materials resistant to strongly alkaline solutions, and is so fitted that a pressure medium can be conveniently admitted into, and bled from the top. Arrangement is also such that a sheet of 9 cm filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is  $7.1 \pm 0.1$  in.<sup>2</sup> ( $4580 \pm 60$  mm<sup>2</sup>). Below the support is a drain tube for discharging the filtrate into a graduated cylinder.

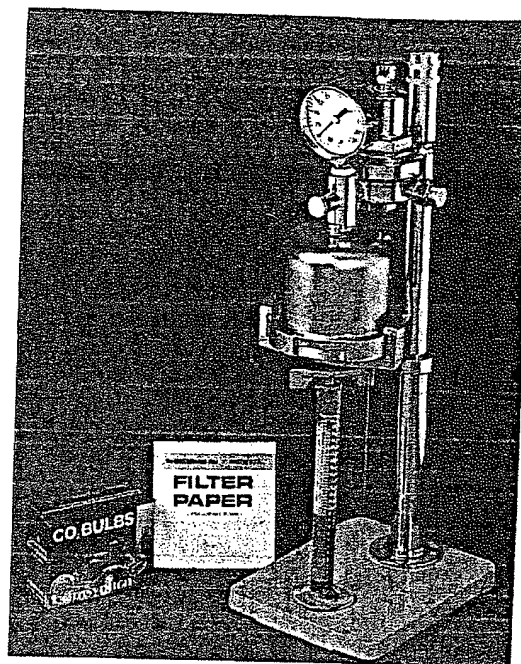
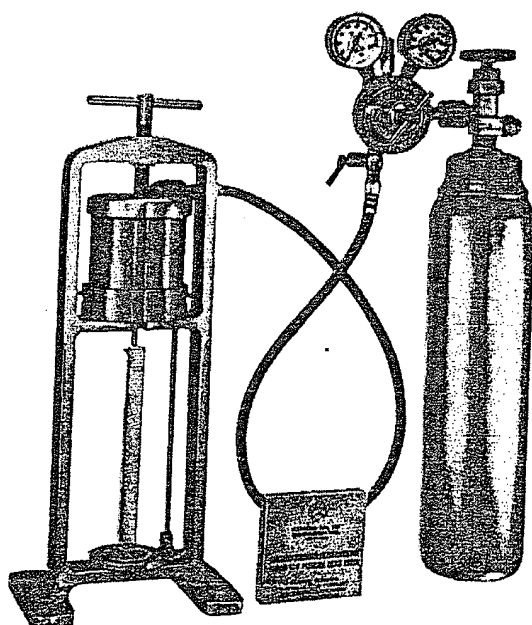
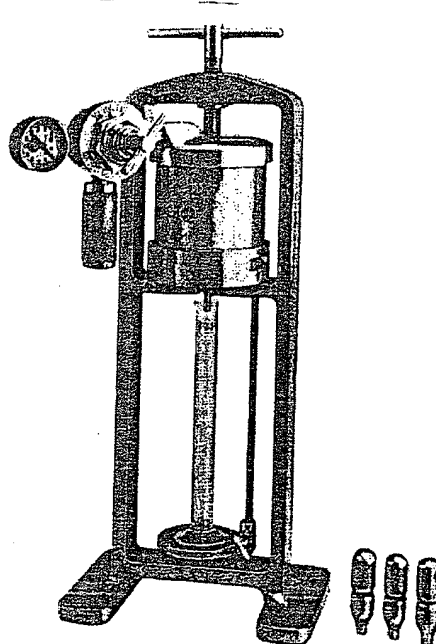


FIG. 3.1  
FILTER PRESS  
With CO<sub>2</sub> Cartridge Pressurization



With Nitrogen Pressurization



With CO<sub>2</sub> Cartridge Pressurization

FIG. 3.2  
FILTER PRESS

Sealing is accomplished with gaskets, and the entire assembly supported by a stand. Figs. 3.1 and 3.2 depict standard units by different manufacturers.

Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midjet pressure cartridges or means for utilizing hydraulic pressure.

To obtain correlative results, one thickness of the proper 90-mm filter paper, Whatman No. 50, S&S No. 576, or equivalent, must be used.

**NOTE:** The mini-press or half-area press does not directly correlate with the results obtained when using the standard sized press.

b. Timer: 30-minute interval

c. Graduated cylinder (TC): 10-cm<sup>3</sup> or 25-cm<sup>3</sup>

### 3.3 Procedure — Low-Temperature/Low-Pressure Test

a. Be sure each part of the cell, particularly the screen, is clean and dry, and that the gaskets are not distorted or worn. Pour the mud sample into the cell to within  $\frac{1}{2}$  in. (13 mm) to top (to minimize CO<sub>2</sub> contamination of filtrate), and complete the assembly with the filter paper in place.

b. Place a dry graduated cylinder under the drain tube to receive the filtrate. Close the relief valve and adjust the regulator so that a pressure of  $100 \pm 5$  psi ( $690 \pm 35$  kPa) is applied in 30 seconds or less. The test period begins at the time of pressure application.

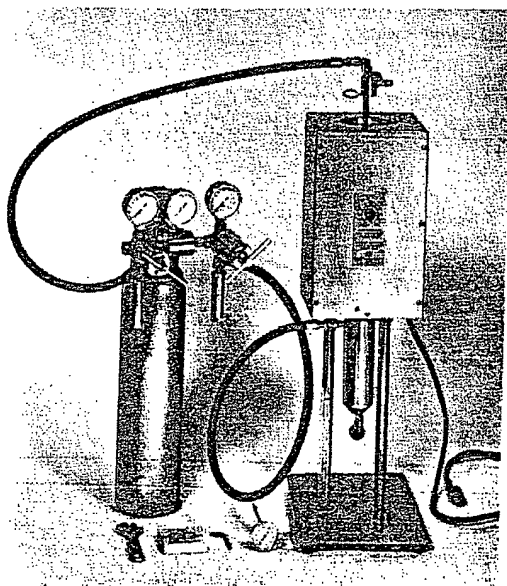


FIG. 3.3  
HIGH TEMPERATURE FILTER PRESS

c. At the end of 30 minutes, measure the volume of filtrate. Shut off the flow through the pressure regulator and open the relief valve carefully. The time interval, if other than 30 minutes, will be reported.

d. Report the volume of filtrate in cubic centimeters (to 0.1 cm<sup>3</sup>) as the API filtrate, and the initial mud temperature in °F (°C). Save the filtrate for appropriate chemical testing.

e. Remove the cell from the frame, first making certain that all pressure has been relieved. Using extreme care to save the filter paper with a minimum of disturbance to the cake, disassemble the cell and discard the mud. Wash the filter cake on the paper with a gentle stream of water.

f. Measure and report the thickness of the filter cake to the nearest  $\frac{1}{16}$  in. (0.8 mm).

g. Although cake descriptions are subjective, such notations as hard, soft, tough, rubbery, firm, etc., may convey important information of cake quality.

### HIGH-TEMPERATURE/HIGH-PRESSURE TEST

#### 3.4 Equipment — High-Temperature/High-Pressure Test

**CAUTION:** All manufacturers' equipment is not capable of the same temperatures and pressures. Rigid adherence to manufacturers' recommendations as to sample volumes, temperatures, and pressures is essential. Failure to do so could result in serious injury.

a. The high-temperature/high-pressure filter press consists of a controlled pressure source (CO<sub>2</sub> or nitro-

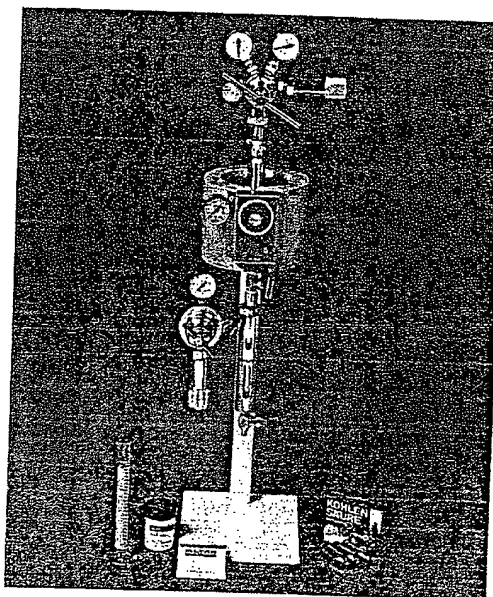


FIG. 3.4  
HIGH TEMPERATURE FILTER PRESS



gen), regulators, a mud cell able to contain working pressures from 600 to 1300 psi, a system for heating the cell, a pressurized collection cell able to maintain proper back pressure (see Table 3.1) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. Figs. 3.3 and 3.4 show available units. The mud cell has a thermometer well, oil-resistant gaskets, a support for the filter medium and a valve on the filtrate delivery tube to control flow from the cell. It may be necessary to replace the gaskets frequently.

**CAUTION:** Nitrous oxide cartridges should not be used as pressure sources for high temperature, high pressure (HTHP) filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

b. Filter medium:

1. Whatman No. 50, or equivalent filter paper, for temperatures to 400°F (204°C).
2. Dynalloy X-5, or equivalent porous disc, for temperatures above 400°F (204°C). A new disc is required for each test.

c. Timer: 30-minute interval

d. Thermometer: up to 500°F (260°C)

e. Graduated cylinder (TC): 25-cm<sup>3</sup> or 50-cm<sup>3</sup>

f. High speed mixer

3.5 Procedure — Temperature to 300°F (149°C)

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain desired temperature.

b. Stir mud sample for 10 minutes with a high speed mixer. Pour the mud sample into the mud cell, being careful not to fill closer than ½ in. (13 mm) from the top to allow for expansion. Install the filter paper.

c. Complete the assembly of the cell and, with both top and bottom valves closed, place it in the heating jacket. Transfer the thermometer to the well in the mud cell.

d. Connect the high-pressure collection cell to the bottom valve and lock in place.

e. Connect a regulated pressure source to the top valve and collection cell, and lock in place.

f. Keeping the valves closed, adjust top and bottom regulators to 100 psi (690 kPa). Open the top valve, applying 100 psi (690 kPa) to the mud. Maintain this pressure until the desired temperature is stabilized. Time of heating of the sample in the filter cell should never exceed a total of one hour.

g. When the sample reaches the selected temperature, increase the pressure of the top pressure unit to 600 psi (4140 kPa) and open the bottom valve to start filtration. Collect the filtrate for 30 minutes, maintaining the selected temperature within ±5°F (±3°C). If back pressure rises above 100 psi (690 kPa) during the test, cautiously reduce the pressure by drawing off a portion of the filtrate. Record the total volume, temperature, pressure and time.

h. The filtrate volume should be corrected to a filter area of 7.1 in.<sup>2</sup> (4580 mm<sup>2</sup>). If the filter area is 3.5 in.<sup>2</sup> (2258 mm<sup>2</sup>), double the filtrate volume and report.

i. At the end of test, close top and bottom valves on mud cell. Bleed pressure from the regulators.

**CAUTION:** Pressure in the mud cell will still be approximately 500 psi (3450 kPa). Keep cell upright and cool to room temperature before disassembling. Bleed pressure from cell before disassembling.

j. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from cell contents and open. Discard mud, and retrieve filter cake. Wash filter cake on the paper with a gentle stream of water.

k. Measure and report the thickness of the filter cake to the nearest 1/32 in. (0.8 mm).

3.6 Procedure — Temperature Above 300°F (149°C)

**CAUTION:** Not all manufacturers' equipment can be used above 300°F (149°C). Know the pressure/temperature rating of equipment in use. Failure to do so could result in serious injury.

Testing at high temperature and high pressure calls for added safety precautions. All pressure cells should be equipped with manual relief valves. Heating jackets should be equipped with both an overheat safety fuse and thermostatic cutoff. Vapor pressure of the liquid phase of muds becomes an increasingly critical design factor as test temperatures are raised. Water vapor pressures at various temperatures are shown in Table 3.1.

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain the correct temperature.

b. Stir mud sample for 10 minutes with a high speed mixer. Pour the mud sample into the mud cell, being careful not to fill the cell closer than 1.5 in. (38 mm) from the top to allow for expansion. Install the proper filter medium (Par. 3.4b).

c. Complete the assembly of the cell, and with top and bottom valves closed, place the mud cell in the heating jacket. Transfer the thermometer to the well in the mud cell.

d. Connect the high-pressure collection cell to the bottom valve, and lock in place.

e. Connect the regulated pressure source to the top valve and the collection cell, and lock in place.

f. With top and bottom valves closed, apply the recommended back pressure (Table 3.1) for the test temperature to both top and bottom. Open the top valve, applying the same pressure to the mud while heating. Maintain this pressure until the test temperature is reached and stabilized.

g. When the temperature of the sample reaches the test temperature, increase the pressure on the top by 500 psi (3450 kPa) over the back pressure being held, and open the bottom valve to begin filtration. Collect the filtrate for 30 minutes, holding the test temperature within  $\pm 5^{\circ}\text{F}$  ( $\pm 3^{\circ}\text{C}$ ) and maintaining the proper back pressure. If the back pressure should begin to rise, it can be reduced by cautiously drawing off a small portion of the filtrate. Time of heating of the sample in the filter cell should never exceed a total of one hour.

h. After the test period close both top and bottom valves on the pressure cell and bleed pressure from the regulators. Allow a minimum of 5 minutes for the filtrate to cool to avoid vaporizing, then cautiously drain and record the total volume. Also record the temperature, pressures, and time. Be sure to allow sufficient time for all the filtrate to drain from the receiver.

**CAUTION:** Pressure inside the filter cell will still be approximately 500 psi (3450 kPa). Keep cell upright and cool to room temperature before disassembly. Bleed pressure from cell before disassembling. Failure to do so could result in serious injury.

i. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from cell contents and open. Discard mud, and retrieve filter cake. Wash filter cake on the paper with a gentle stream of water.

j. Measure and report the thickness of the filter cake to the nearest  $\frac{1}{16}$  in. (0.8 mm).

TABLE 3.1  
RECOMMENDED MINIMUM BACK PRESSURE

Test Temperature		Vapor Pressure		Minimum Back Pressure	
$^{\circ}\text{F}$	$^{\circ}\text{C}$	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
..... LIMIT of "Normal" Field Testing .....					
*350	177	135	932	160	1104
*400	204	247	1704	275	1898
*450	232	422	2912	450	3105

\*Do not exceed equipment manufacturers' recommendations for maximum temperatures, pressures, and volumes.



## SECTION 4 WATER, OIL AND SOLIDS

**4.1 Description.** The retort instrument provides a means for separating and measuring the volumes of water, oil and solids contained in a sample of water-based mud. In the retort, a known volume of a whole water mud sample is heated to vaporize the liquid components which are then condensed and collected in a graduated receiver. Liquid volumes are determined directly from reading the oil and water phases in the receiver. Total volume of solids (suspended and dissolved) is obtained by difference (total sample volume — liquid volume). Calculations are necessary to determine the volume of suspended solids since any dissolved solids will be retained in the retort. The relative volumes of low gravity solids and weighting material can also be calculated. Knowledge of the solids concentration and composition is considered basic to viscosity and filtration control in water-based muds.

### 4.2 Equipment

- a. Retort instrument (Figs. 4.1 and 4.2)  
Retorts of two sizes (10-cm<sup>3</sup> and 20-cm<sup>3</sup>) are commonly available. Specifications of these retorts are:

#### 1. Sample cup

	Sample Cup Size	
	10-cm <sup>3</sup>	20-cm <sup>3</sup>
Total volume	10-cm <sup>3</sup>	20-cm <sup>3</sup>
Precision	±0.05 cm <sup>3</sup>	±0.10 cm <sup>3</sup>

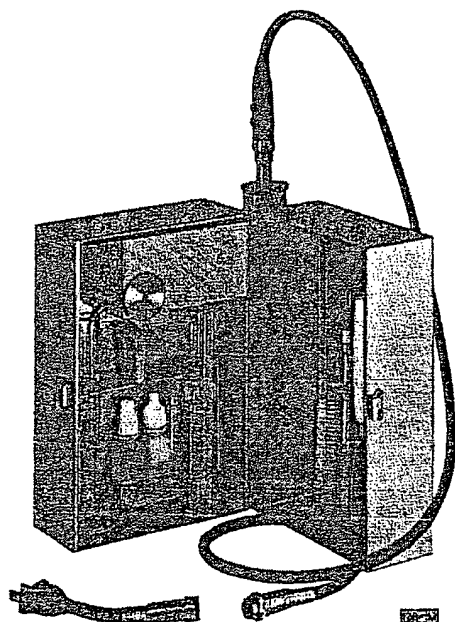


FIG. 4.1  
RETORT FOR LIQUID AND SOLID CONTENT

2. Liquid condenser — sufficient mass to cool the oil and water vapors below their vaporization temperature prior to leaving the condenser.
3. Heating element — sufficient wattage to raise the temperature of the sample above the vaporization point of the liquid components within 15 minutes without causing solids boil-over.
4. Temperature control — a temperature control is desirable. It should be capable of limiting the temperature of the retort to  $930 \pm 70^\circ\text{F}$  ( $500 \pm 20^\circ\text{C}$ ).

#### b. Liquid receiver

1. Graduated cylinder, or tube
2. Material — transparent and inert to oil, water, salt solution and temperatures up to  $90^\circ\text{F}$  ( $32^\circ\text{C}$ ).

#### 3. Specifications of graduation marks:

Precision	Graduate Size	
	10-cm <sup>3</sup>	20-cm <sup>3</sup>
	±0.10 cm <sup>3</sup>	±0.20 cm <sup>3</sup>
Graduation	0.10 cm <sup>3</sup>	0.20 cm <sup>3</sup>
Calibration method for marks	TC (to contain at $20^\circ\text{C}$ )	
Scale to read	cm <sup>3</sup> and/or volume percent	



FIG. 4.2  
RETORT FOR LIQUID AND SOLID CONTENT

- c. Fine steel wool (i.e., No. 000)

NOTE: "Liquid Steel Wool" or similar products are not recommended for this application.

- d. High temperature silicone grease. (Used for a thread seal and a lubricant.)

- e. Pipe cleaners

- f. Putty knife or spatula with blade shaped to fit the inside dimensions of the sample cup of the retort.

- g. Defoaming agent

#### 4.3 Procedure

NOTE: Procedure will vary slightly depending on type of retort used. See manufacturer's instructions for complete procedure.

- a. Be sure the retort sample cup, condenser passage and liquid receiver are clean, dry and cooled from previous use.

1. The inside of the sample cup and lid must be thoroughly cleaned prior to each test. Periodically, the interior of the sample cup should also be lightly polished with steel wool.

2. The condenser passage should also be cleaned and dried before each test using pipe cleaners. A build-up of material in the condenser can decrease condensation efficiency and cause erroneous liquid readings in a test.

- b. Collect a representative sample of water-based mud and allow it to cool to approximately 80°F (26°C). Screen the sample through the 20-mesh screen on the Marsh Funnel to remove lost circulation material, large cuttings or debris.

- c. If mud sample contains gas or air, add 2 to 3 drops of defoaming agent to about 300 cm<sup>3</sup> of mud and stir slowly for 2 to 3 minutes to release gases.

- d. Lubricate the threads on the sample cup and condenser tube with a light coating of silicone grease. This prevents vapor loss through the threads and also facilitates disassembly of the equipment and clean-up at the end of the test.

- e. Lightly pack a ring of steel wool into the chamber above the sample cup. Use only enough steel wool to prevent boil over of solids into the liquid receiver. (This is determined from experience.)

- f. Fill the retort sample cup with gas-free water mud, from Par. 4.3c.

- g. Carefully place the lid on the sample cup and allow an overflow of the sample through the hole in the lid to insure that the correct volume of sample is in the cup.

- h. With the lid held tightly in place, wipe the overflow from the sample cup and lid. Be sure that the sample cup threads are still covered with silicone grease after wiping, and that the hole in the lid is not plugged.

- i. Screw the retort cup onto the retort chamber with its condenser.

- j. Place a clean, dry, liquid receiver under the condenser discharge tube.

- k. Heat the retort and observe the liquid falling from the condenser. Continue heating for ten minutes beyond the time that no more condensate is being collected.

- l. Remove the liquid receiver from the retort. Notice if solids are in the liquid which was recovered. If so, whole mud has boiled over from the sample cup and the test must be repeated from Par. 4.3a.

- m. Read the volumes of water and oil in liquid receiver after it has cooled to ambient temperature. Record the volumes (or volume percentage) of water and oil collected.

#### 4.4 Calculation

Using the measured volumes of oil and water and the volume of the original whole mud sample (10-cm<sup>3</sup> or 20-cm<sup>3</sup>), calculate the volume percent water, oil and total solids in the mud.

Volume percent water:

$$V_w = \frac{100 (\text{volume of water, cm}^3)}{\text{volume of sample, cm}^3} \quad (a)$$

Volume percent oil:

$$V_o = \frac{100 (\text{volume of oil, cm}^3)}{\text{volume of sample, cm}^3} \quad (b)$$

Volume percent retort solids:

$$V_s = 100 - (V_w + V_o) \quad (c)$$

NOTE: The volume percent retort solids above is only the difference between water plus oil, and the total sample volume (10-cm<sup>3</sup> or 20-cm<sup>3</sup>). This difference is both suspended solids (weighting material and low gravity) and dissolved materials (e.g., salt). This volume percent retort solids is the suspended solids only if the mud is an untreated, fresh water mud.

- b. Additional calculations are required to find the volume percent suspended solids and relate them to the relative volumes of low gravity solids and weighting material. To make these calculations, an accurate mud weight and chloride concentration are needed.

$$V_{ss} = V_s - V_w \frac{C_s}{1.680,000 - 1.21 C_s} \quad (d)$$

where:

$V_{ss}$  = volume percent suspended solids

$C_s$  = chloride concentration, mg/L

Volume percent low gravity solids are calculated as:

$$V_{lg} = \frac{1}{(\rho_b - \rho_g)} [100\rho_f + (\rho_b - \rho_f)V_{ss} - 12W_m - (\rho_f - \rho_b)V_o] \quad (e)$$

where:

$V_{lg}$  = volume percent low gravity solids

$W_m$  = mud weight, lb/gal

$\rho_f$  = density of filtrate, g/cm<sup>3</sup>

$(\rho_f = 1 + 0.00000109 C_s)$  based on sodium chloride (f)

$\rho_b$  = density of weighting material, g/cm<sup>3</sup>

$\rho_{lg}$  = density of low gravity solids, g/cm<sup>3</sup>  
(use 2.6 if unknown)

$\rho_o$  = density of oil, g/cm<sup>3</sup> (use 0.84 if unknown)

Volume percent weighting material ( $V_b$ ) is calculated as:

$$V_b = V_{ss} - V_{lg}$$

Concentration of low gravity solids, weighting material and suspended solids can be calculated as:

$$C_{lg} = 3.49 (\rho_{lg}) (V_{lg}) \quad (h)$$

$$C_b = 3.49 (\rho_b) (V_b) \quad (i)$$

$$C_{ss} = C_{lg} + C_b \quad (j)$$

where:

$C_{lg}$  = low gravity solids concentration, lb/bbl

$C_b$  = weighting material concentration, lb/bbl

$C_{ss}$  = suspended solids concentration, lb/bbl

## SECTION 5 SAND

5.1 Description. The sand content of mud is the volume percent of particles larger than 74 microns. It is measured by a sand-screen set (see Fig. 5.1).

### 5.2 Equipment

- a. 200-mesh sieve, 2.5 in. (63.5 mm) in diameter
- b. Funnel to fit sieve
- c. Glass measuring tube marked for the volume of mud to be added. The tube is graduated from 0 to 20 percent in order to read directly the percentage of sand.

### 5.3 Procedure

- a. Fill the glass measuring tube with mud to the "mud" mark. Add water to the next mark. Close the mouth of the tube and shake vigorously.
- b. Pour the mixture onto the clean, wet screen. Discard the liquid passing through the screen. Add more water to the tube, shake, and again pour onto the screen. Repeat until the tube is clean. Wash the sand retained on the screen to free it of any remaining mud.
- c. Put the funnel upside down over the top of the sieve. Slowly invert the assembly and insert the tip of the funnel into the mouth of the glass tube. Wash the sand into the tube by playing a fine spray of water through the screen. Allow the sand to settle. From the graduations on the tube, read the volume percent of the sand.
- d. Report the sand content of the mud in volume percent. Report the source of the mud sample, i.e., above shaker, suction pit, etc. Coarse solids other than sand will be retained on the screen (e.g., lost circulation material) and the presence of such solids should be noted.



FIG. 5.1  
SAND-CONTENT SET

## SECTION 6 METHYLENE BLUE CAPACITY

### METHYLENE BLUE CAPACITY OF DRILLING FLUIDS

#### 6.1 Description

a. The methylene blue capacity of drilling fluid is an indication of the amount of reactive clays (bentonite and/or drill solids) present as determined by the methylene blue test (MBT). The methylene blue capacity provides an estimate of the total cation exchange capacity (CEC) of the drilling fluid solids. Methylene blue capacity and cation exchange capacity are not necessarily equivalent, the former normally being somewhat less than the actual cation exchange capacity.

b. Methylene blue solution is added to a sample of drilling fluid (which has been treated with hydrogen peroxide and acidified) until saturation is noted by formation of a dye "halo" around a drop of solids suspension placed on filter paper. Variations of the procedure used on the drilling fluid can be performed on drill solids and commercial bentonite to allow an estimate of the amount of each type of solid present in the fluid (see API RP 13I, Section 9).

c. Drilling fluids frequently contain substances in addition to reactive clays that adsorb methylene blue. Pretreatment with hydrogen peroxide (see Par. 6.3b) is intended to remove the effect of organic materials such as lignosulfonates, lignites, cellulosic polymers, polyacrylates, etc.

#### 6.2 Equipment

a. Methylene blue solution: 3.20 g Reagent grade methylene blue ( $C_{16}H_{18}N_3SCl$ )/L (1 cm<sup>3</sup> = 0.01 milliequivalent) (CAS # 61-73-4)

**NOTE:** The moisture content of Reagent grade methylene blue must be determined each time the solution is prepared. Dry a 1.000 gram portion of methylene blue to a constant weight at  $200 \pm 5^\circ F$  ( $93 \pm 3^\circ C$ ). Make the appropriate correction in the weight of methylene blue to be taken to prepare the solution as follows:

$$\frac{\text{Weight of sample to be taken, g}}{\text{weight of dried sample}} = \frac{3.20}{\text{weight of dried sample}} \quad (a)$$

b. Hydrogen peroxide: 3 percent solution. (CAS #7722-88-5)

c. Dilute sulfuric acid: approximately 5N

d. Syringe (TD): 2.5-cm<sup>3</sup> or 3-cm<sup>3</sup>

e. Erlenmeyer flask: 250-cm<sup>3</sup>

f. Burette (TD): 10-cm<sup>3</sup>, micropipette: 0.5-cm<sup>3</sup>, or graduated pipette: 1-cm<sup>3</sup>

g. Graduated cylinder (TD): 50-cm<sup>3</sup>

h. Stirring rod

i. Hot plate

j. Whatman No. 1 filter paper, or equivalent

#### 6.3 Procedure

a. Add 2.0 cm<sup>3</sup> of drilling fluid (or suitable volume of drilling fluid to require from 2-10 cm<sup>3</sup> of methylene blue solution) to 10 cm<sup>3</sup> of water in the Erlenmeyer flask. To assure that exactly 2.0 cm<sup>3</sup> is being added, use the following procedure:

1. The syringe should have a capacity of more than 2 cm<sup>3</sup>, generally 2.5 or 3 cm<sup>3</sup>. By using a larger syringe, it is not necessary to remove the air trapped in the syringe.

2. The air or gas entrained in the drilling fluid must be removed. (See Appendix D.) Stir the drilling fluid to break the gel and quickly draw the drilling fluid into the syringe. Then slowly discharge the syringe back into the drilling fluid keeping the tip submerged.

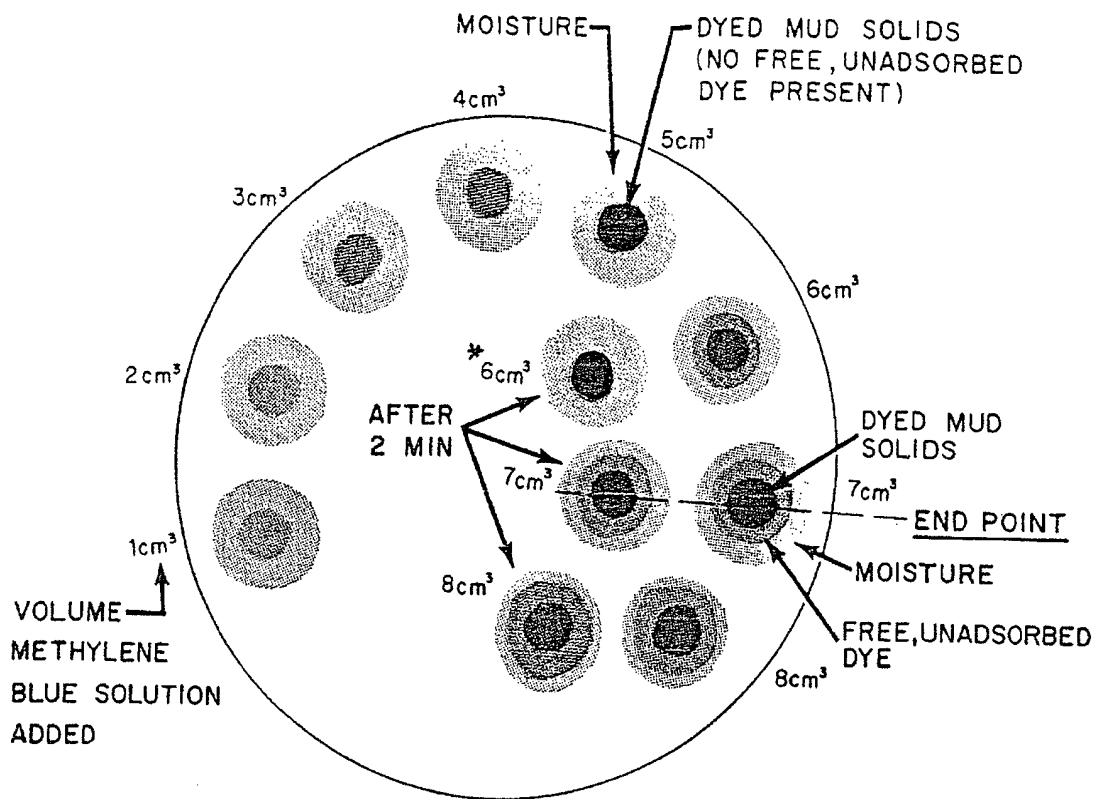
3. Again draw the drilling fluid into the syringe until the end of the plunger is at the last graduation on the syringe (e.g., at the 3 cm<sup>3</sup> line on a 3 cm<sup>3</sup> syringe.)

4. Deliver 2.0 cm<sup>3</sup> of drilling fluid by pushing the plunger until the end of the plunger is exactly 2 cm<sup>3</sup> from the last graduation on the syringe. Thus in a 3 cm<sup>3</sup> syringe, it would be at the 1 cm<sup>3</sup> line.

b. Add 15 cm<sup>3</sup> of 3 percent hydrogen peroxide and 0.5 cm<sup>3</sup> of sulfuric acid. Boil gently for 10 minutes, but do not allow to boil to dryness. Dilute to about 50 cm<sup>3</sup> with water.

c. Add methylene blue to the flask in increments of 0.5 cm<sup>3</sup>. If the approximate amount of methylene blue solution necessary to reach the endpoint is known from previous testing, larger increments (1-2 cm<sup>3</sup>) can be used at the beginning of the titration. After each addition of methylene blue solution, swirl the contents of the flask for about 30 seconds. While the solids are still suspended, remove one drop of liquid with the stirring rod and place the drop on the filter paper. The initial endpoint of the titration is reached when dye appears as a blue or turquoise ring surrounding the dyed solids as shown in Fig. 6.1.

d. When the blue tint spreading from the spot is detected, shake the flask an additional 2 minutes and place another drop on the filter paper. If the blue ring is again evident, the final endpoint has been reached. If the blue ring does not appear, continue as before (Par. 6.3c) until a drop taken after 2 minutes shows the blue tint.



\*Note: Free dye detected immediately after adding sixth  $\text{cm}^3$  is adsorbed after two minutes and indicates that end point has not quite been reached.

FIG. 6.1  
SPOT TESTS FOR END POINT OF METHYLENE BLUE TITRATION

6.4 Calculation. Report the methylene blue capacity (MBT) of the drilling fluid, calculated as follows:

$$\text{Methylene blue capacity, cm}^3/\text{cm}^3 = \frac{\text{methylene blue, cm}^3}{\text{drilling fluid, cm}^3} \quad (a)$$

Alternately, the methylene blue capacity can be reported as lb/bbl bentonite equivalent (based on bentonite with a cation exchange capacity of 70 meq/100 g) calculated as follows:

$$\text{Bentonite equivalent, lb/bbl} = \frac{5 (\text{methylene blue, cm}^3)}{\text{drilling fluid, cm}^3} \quad (b)$$

$$\text{Bentonite equivalent, kg/m}^3\text{g,} = \frac{2.85 (\text{bentonite equivalent, lb/bbl})}{1} \quad (c)$$

NOTE: The lb/bbl bentonite equivalent from (b or c) is not equal to the amount of commercial bentonite in the drilling fluid. Reactive clays in the drill solids contribute to this quantity as well as commercial bentonite. See API RP 13I Section 9 for additional information on estimating the amount of commercial bentonite and drill solids present.

## SECTION 7

### pH

#### 7.1 Description

a. Field measurement of drilling fluid (or filtrate) pH and adjustments to the pH are fundamental to drilling fluid control. Clay interactions, solubility of various components and contaminants, and effectiveness of additives are all dependent on pH, as is the control of acidic and sulfide corrosion processes.

b. The term "pH" denotes the negative logarithm of the hydrogen ion,  $H^+$ , activity in aqueous solutions (activity and concentration are equal only in dilute solutions):

$$pH = -\log [H^+]$$

For pure water at 75°F (24°C) the hydrogen ion activity  $[H^+]$  is  $10^{-7}$  moles/liter and  $pH = 7$ . This system is termed "neutral" because the hydroxyl ion activity  $[OH^-]$  is also  $10^{-7}$  moles/liter. In aqueous systems at 24°C the ion product,  $[H^+] \times [OH^-]$ , is  $10^{-14}$  (a constant). Consequently, an increase in  $H^+$  denotes a like decrease in  $[OH^-]$ . A change in pH of one unit indicates a ten-fold change in both  $[H^+]$  and  $[OH^-]$ . Solutions with pH less than 7 are termed "acidic" and those with pH greater than 7 are termed "basic" or "alkaline."

c. The recommended method for pH measurement of drilling fluid is with a glass electrode pH meter. This method is accurate and gives reliable pH values, being free of interferences if a high quality electrode system is used with a properly designed instrument. Rugged pH instruments are available that automatically temperature compensate the slope and are preferred over the manually adjusted instruments.

**NOTE:** Color matching pH paper and sticks are used for field pH measurements, but are not the methods recommended. These methods are reliable only in very simple water muds. Mud solids, dissolved salts and chemicals, and dark-colored liquids cause serious errors in pH paper values. Readability is normally about 0.5 pH unit.

#### 7.2 Equipment

a. pH meter: millivolt range potentiometer calibrated to show pH units for measuring the potential between a glass-membrane electrode and a standard "reference" electrode. The instrument is (preferred) to be water, shock, and corrosion-resistant and portable. Specifications are:

1. pH range: 0 to 14
2. Electronics type: solid state (preferred)
3. Power Source: batteries (preferred)
4. Operating temperature range: 32-150°F (0-66°C)
5. Readout: digital (preferred)

6. Resolution: 0.1 pH unit

7. Accuracy:  $\pm 0.1$  pH unit

8. Repeatability: 0.1 pH unit

9. Adjustments

(a) "temperature" compensation of electrode system

(b) "slope" of electrode system (preferred)

(c) "calibration" setting of readout. (Instrument with the above internal temperature compensation is preferred).

b. Electrode system: A combination system of a glass electrode for sensing  $H^+$  ions and a standard voltage reference electrode, constructed as a single electrode (preferred). Body of this probe should be constructed of durable material. A flat-end probe is preferred for better protection and easier cleaning of the electrode. Waterproof connection to the meter is recommended. Specifications are:

1. Glass pH electrode response range: 0 to 14 pH unit

2. Electrodes: a glass electrode and a silver/silver chloride electrode in combination, having a ceramic or a plastic single or double junction.

**NOTE:** Use double junction electrode for measuring liquids containing sulfide or bromide ion to avoid damaging (silver) reference electrode system.

3. Electrolyte in reference electrode: KCl gel

4. Glass composition: suitable for low sodium ion error.

5. Sodium ion error: at  $pH = 13$  or at 0.1 mole  $Na^+$  ion, an error less than 0.1 pH unit.

c. Buffer solutions: three solutions to calibrate and set slope of pH meter prior to sample measurement.

1.  $pH = 4.0$ : potassium hydrogen phthalate at 0.05 molar in water. Gives 4.01 pH at 75°F (24°C).

2.  $pH = 7.0$ : potassium dihydrogen phosphate at 0.02066 molar and disodium hydrogen phosphate at 0.02934 molar in water. Gives 7.00 pH at 75°F (24°C).

3.  $pH = 10.0$ : sodium carbonate at 0.025 molar and sodium bicarbonate at 0.025 molar in water. Gives 10.01 pH at 75°F (24°C).

**NOTE:** Buffers may be obtained from supply houses as pre-made solution, dry-powder packages, or a given formula, but must duplicate National Bureau of Standards primary or secondary buffers. Shelf life of all buffers not to exceed six months

before disposal. Date of preparation of buffer should be shown on bottles used in the field. Bottles should be kept tightly stoppered.

- d. Distilled or deionized water: in spray bottle
- e. Soft tissues: to blot electrodes
- f. Thermometer: glass, 32-220°F (0-150°C)
- g. Accessory equipment:
  - 1. Soft-bristle test tube brush: to clean electrode
  - 2. Mild liquid detergent: Ivory®, or equivalent
  - 3. Electrode storage vial: to keep electrodes moist
  - 4. Sodium Hydroxide: 0.1 molar (approximately); to recondition electrode.
  - 5. Hydrochloric Acid: 0.1 molar (approximately); to recondition electrode.
  - 6. Ammonium Bifluoride: 10% solution (approximately); to recondition electrode.  
CAUTION: This is a strong and toxic acid.
  - 7. Hydrofluoric acid: ACS reagent grade.  
CAUTION: This is a strong acid.

### 7.3 Procedure — pH Measurement

- a. Obtain sample of fluid to be tested. Allow it to reach  $75 \pm 5^\circ\text{F}$  ( $24 \pm 3^\circ\text{C}$ ).
- b. Allow buffer solution to also reach the same temperature as the fluid to be tested.

NOTE: For accurate pH measurement the test fluid, buffer solution, and reference electrode must all be at the sample temperature. The pH of the buffer solution indicated on the container label is the correct pH only at  $75^\circ\text{F}$  ( $24^\circ\text{C}$ ). If attempting to calibrate at another temperature, the actual pH of the buffer at this temperature must be used. Tables of buffer pH values at various temperatures are available from the suppliers and should be used in the calibration procedure.

- c. Clean electrodes by washing with distilled water and blot dry.
- d. Place probe into pH 7.0 buffer.
- e. Turn on meter; wait 60 seconds for reading to stabilize. (See Par. 7.4a if meter reading is not stable.)
- f. Measure temperature of pH 7 buffer solution.
- g. Set this temperature on "temperature" knob.
- h. Set meter reading to "7.0" using "calibration" knob.
- i. Rinse probe with distilled water and blot dry.

j. Repeat operations in Par. 7.3f through 7.3i using either pH 4.0 or pH 10.0 buffer. Use pH 4.0 if "acidic" sample, or pH 10.0 if "alkaline" sample is to be tested. Set meter to number "4.0" or "10.0" respectively, using "slope" adjustment knob. (If no "slope" knob exists, use the "temperature" knob to set "4.0" or "10.0" on meter.)

k. Check the meter with pH 7 buffer again. If it has changed, reset to "7.0" with "calibration" knob. Repeat Par. 7.3f through 7.3k. If meter does not calibrate properly, recondition or replace electrodes as given in Par. 7.4a through 7.4f.

NOTE: Discard and do not reuse the sample of buffer solutions used in calibration. Meter should be fully calibrated every day, Par. 7.3b through 7.3k, using two buffers. Check with pH 7 buffer every three hours.

l. If meter calibrates properly, rinse electrode with distilled water and blot dry. Place electrode in sample to be tested and stir gently. Allow 60 to 90 seconds for reading to stabilize.

m. Record sample pH to nearest 0.1 pH unit and the temperature of sample tested on Drilling Mud Report form.

n. Carefully clean the electrode in preparation for next usage. Store in vial of pH 4 buffer. NEVER let the probe tip become dry.

o. Turn meter off and close cover to protect instrument. Avoid storing instrument at extreme temperatures. (Below  $32^\circ\text{F}$  ( $0^\circ\text{C}$ ) or above  $120^\circ\text{F}$  ( $49^\circ\text{C}$ )).

### 7.4 Procedure — Care of Electrode

a. Cleaning the electrode will be necessary periodically, especially if oil or clay particles coat the face of the glass electrode or the porous frit of the reference electrode. Clean electrode with a soft-bristle brush and a mild detergent.

b. Reconditioning the electrode may be necessary if plugging becomes severe, as indicated by slow response, drifting of readings, or if "slope" and "calibration" cannot be mutually set.

c. Recondition by soaking electrode for 10 minutes in 0.1 M HCl followed by rinsing in water and soaking for 10 minutes in 0.1 M NaOH and rinsing again.

d. Check electrode for response by performing calibration Par. 7.3b through 7.3k.

e. If electrode continues to perform poorly, soak electrode for two minutes only in 10%  $\text{NH}_4\text{F} \cdot \text{HF}$  solution. (CAUTION: This is strong and toxic acid.) Repeat Par. 7.3b through 7.3k to check for calibration capability.

f. Replace electrode system if above steps fail to recondition it.



## SECTION 8 CHEMICAL ANALYSIS

### ALKALINITY AND LIME CONTENT

#### 8.1 Description

a. Alkalinity can be considered as the acid-neutralizing power of a substance. In drilling fluid testing, alkalinity measurements can be made on either the whole mud (designated with a subscript m) or on the filtrate (designated with a subscript f). The data collected from the alkalinity test can also be used to estimate the concentrations of hydroxyl ( $\text{OH}^-$ ), carbonate ( $\text{CO}_3^{2-}$ ) and bicarbonate ( $\text{HCO}_3^-$ ) ions in the drilling fluid.

b. Knowledge of the mud and filtrate alkalinities is important in many drilling operations to insure proper control of the mud chemistry. Mud additives, particularly some deflocculants, require an alkaline environment to function properly. The source and nature of the alkalinity exists. Alkalinity arising from hydroxyl ions is generally accepted as being beneficial while alkalinities resulting from carbonates and/or bicarbonates may have adverse effects on the mud performance.

c. The ions that are primarily responsible for filtrate alkalinities are the hydroxyl ( $\text{OH}^-$ ), carbonate ( $\text{CO}_3^{2-}$ ), and bicarbonate ( $\text{HCO}_3^-$ ) ions. It is important to realize that the carbonate species can change from one form to another form by changing the solution pH. The interpretation of filtrate alkalinities involves calculating differences between the titration values obtained by the following procedures. It is for this reason that special attention to accurate measurement of the various reagents is important in all steps of the procedure. In addition it is important to realize that the following calculations are only estimates of the concentrations of the reported ionic species based on theoretical chemical equilibrium reactions:

d. The composition of mud filtrates is often so complex that the interpretation of alkalinities in terms of estimated ionic components may be misleading. Any particular alkalinity value represents all of the ions which will react with the acid in the pH range over which that particular value was tested. Inorganic ions which may contribute to the alkalinity, in addition to the hydroxyl, carbonate, and bicarbonate ions, are: borates, silicates, sulfides, and phosphates. Perhaps more serious in drilling fluids, and anionic organic thinners, filtrate reducers, and their degradation products may contribute to a large portion of the alkalinity value as well as masking the endpoint color change. These organic materials make a particularly large contribution to the  $M_f$  alkalinity and thus render the test highly inaccurate in muds treated with organic thinners. However, for simple bentonite base mud systems containing no organic thinners the  $P_f$  and  $M_f$  alkalinities can be used as guidelines to determine both the presence of carbonate/bicarbonate contamination and the treatment necessary to alleviate the contamination. If organic thinners are present in large amounts, the  $P_f/M_f$  test is suspect and the  $P_f/P_m$  method should be used.

#### 8.2 Equipment

a. Sulfuric acid solution: standardized 0.02 Normal (N/50) (CAS #7664-93-9)

b. Phenolphthalein indicator solution: 1 g/100 cm<sup>3</sup> of a 50% alcohol/water solution (CAS #518-51-4)

c. Methyl orange indicator solution: 0.1 g/100 cm<sup>3</sup> of water (CAS #547-58-0)

d. pH Meter (optional)

**NOTE:** pH meter is more accurate than indicator solution.

e. Titration vessel: 100-150-cm<sup>3</sup>, preferably white

f. Serological (graduated) pipettes (TD): one 1-cm<sup>3</sup> and one 10-cm<sup>3</sup>.

g. Volumetric pipette (TD): 1-cm<sup>3</sup>

h. Hypodermic syringe (TD): 1-cm<sup>3</sup>

i. Stirring rod

#### 8.3 Procedure — Filtrate Alkalinity: $P_f$ , $M_f$

a. Measure one or more cm<sup>3</sup> of filtrate into the titration vessel. Add two or more drops of the phenolphthalein indicator solution. If the indicator turns pink, add 0.02 normal (N/50) sulfuric acid, drop by drop from the graduated pipette, while stirring, until the pink color just disappears. If the sample is so colored that the indicator color change is masked, the endpoint can be taken when the pH drops to 8.3, as measured with a pH meter. (Refer to Section 7 for proper pH measurement.)

b. Report the phenolphthalein alkalinity of the filtrate,  $P_f$ , as the number of cm<sup>3</sup> of 0.02 normal acid required per cm<sup>3</sup> of filtrate.

c. To the sample which has been titrated to the  $P_f$  endpoint, add two or three drops of methyl orange indicator solution. Add the standard acid drop by drop from the pipette, while stirring, until the color of the indicator changes from yellow to pink. The endpoint can also be taken when the pH of the sample drops to 4.3 as measured by a pH meter. (Refer to Section 7 for proper pH measurement.)

d. Report the methyl orange alkalinity of the filtrate,  $M_f$ , as the total cm<sup>3</sup> of 0.02 normal acid per cm<sup>3</sup> of filtrate required to reach the methyl orange endpoint (including that amount required for the  $P_f$  endpoint.)

#### 8.4 Procedure — Mud Alkalinity: $P_m$

a. Measure 1.0 cm<sup>3</sup> of mud into the titration vessel using a syringe or volumetric pipette. Dilute the mud sample with 25-50 cm<sup>3</sup> of distilled water. Add 4-5 drops of phenolphthalein indicator solution and, while stirring, titrate rapidly with 0.02 normal (N/50) standard sulfuric acid solution until the pink color disappears. If the endpoint color change cannot be seen, it can be taken when the pH drops to 8.3 measured by a pH meter. (Refer to Section 7 for proper pH measurement.)

**NOTE:** If cement contamination is suspected, the titration must be performed as rapidly as possible and the endpoint reported as the first disappearance of the pink color.

b. Report the phenolphthalein alkalinity of the mud,  $P_m$ , as the number of  $\text{cm}^3$  of 0.02 normal (N/50) acid required per  $\text{cm}^3$  of mud.

**8.5 Calculation —  $P_f$ ,  $M_f$ .** The concentrations of hydroxyl, carbonate, and bicarbonate ions can be estimated as follows:

**TABLE 8.1**  
CONCENTRATIONS, mg/L

	$\text{OH}^-$	$\text{CO}_3^{2-}$	$\text{HCO}_3^-$
$P_f = 0$	0	0	$1220M_f$
$2P_f < M_f$	0	$1200P_f$	$1220(M_f - 2P_f)$
$2P_f = M_f$	0	$1200P_f$	0
$2P_f > M_f$	$340(2P_f - M_f)$	$1200(M_f - P_f)$	0
$P_f = M_f$	$340M_f$	0	0

#### 8.6 Procedure — Estimated Lime Content

a. Determine the  $P_f$  and  $P_m$  of the filtrate and mud as described in Pars. 8.3 and 8.4.

b. Determine the volume fraction of water in the mud,  $F_w$ , using the value for volume percent water from the liquid and solids determination (Section 4) in the following equation:

$$F_w = \frac{\text{Volume percent water}}{100} \quad (a)$$

**8.7 Calculation — Estimated Lime Content.** Report the lime content of the mud in pounds per barrel (lb/bbl) from the following equation:

$$\text{Estimated Lime, lb/bbl} = 0.26 (P_m - F_w P_f) \quad (a)$$

$$\text{Estimated Lime, kg/m}^3 = 0.742 (P_m - F_w P_f) \quad (b)$$

#### ALTERNATE ALKALINITY METHOD

**8.8 Description.** The P1/P2 back-titration method was mainly developed in an attempt to overcome the limitations of the  $P_f/M_f$  alkalinity method. (Par. 8.1 through 8.5.) The P1/P2 method also has limitations. A comparison of the generally accepted advantages and disadvantages of the two alkalinity measurements are listed in Table 8.2.

#### 8.9 Equipment — P1/P2 Alkalinity Method

a. Hydrochloric acid solution: standardized 0.02 normal (N/50) (CAS #7647-01-0)

b. Sodium hydroxide solution: 0.1 normal (N/10) (CAS #1310-73-2)

c. Barium chloride solution: 10 percent, neutralized to pH 7 with NaOH (CAS # 10361-37-2)

d. Phenolphthalein indicator solution: 1 g/100  $\text{cm}^3$  of a 50% alcohol-water solution (CAS #518-51-4)

e. Deionized water

f. pH paper strips: 6-12 range

**TABLE 8.2**  
FILTRATE ALKALINITY METHODS  
COMPARISON

Method	Advantage	Disadvantage
$P_f/M_f$	a. Traditional method	a. Interference in the $M_f$ titration
	b. 2 titrations, 1 sample	b. Bicarbonate result normally too high
P1/P2	a. Eliminates interference in $M_f$ titrations	a. 3 titrations with 3 samples
		b. Caustic measurement critical
		c. Uses a toxic material ( $\text{BaCl}_2$ )

**NOTE:** The total carbonate concentration in a drilling fluid can also be determined by use of the Garrett Gas Train as described in the Appendix A, Carbonate.

g. pH meter, optional (as discussed in Section 7)

h. Titration vessel: 100-150- $\text{cm}^3$ , preferably white

i. Volumetric pipette (TD): one 1- $\text{cm}^3$  and one 2- $\text{cm}^3$

j. Burette (TD): automatic student type, 25- $\text{cm}^3$

k. Graduated cylinders (TC): one 25- $\text{cm}^3$  and one 5- $\text{cm}^3$  or 10- $\text{cm}^3$

l. Stirring rod

#### 8.10 Procedure — P1/P2 Alkalinity Method

a. Determine the  $P_f$  alkalinity as described in Par. 8.2. 8.3a and 8.3b.

b. Using a volumetric pipette, measure 1.0  $\text{cm}^3$  of filtrate into the titration vessel. Add 25  $\text{cm}^3$  of deionized water to the titration vessel.

c. Using a volumetric pipette, add 2.0  $\text{cm}^3$  of 0.1 normal (N/10) sodium hydroxide solution and stir well. Measure the pH with the high range pH paper (or pH meter). If the pH is 11.4 or greater, proceed to Par. 8.10d. If the pH is less than 11.4 add 2.0  $\text{cm}^3$  more of 0.1 normal sodium hydroxide solution, then proceed to Par. 8.10d.

**NOTE:** Exact measurement of the sodium hydroxide is necessary to avoid serious errors.

d. Using the small graduated cylinder, measure 3  $\text{cm}^3$  of barium chloride and add to the titration vessel. Add 2-4 drops of phenolphthalein indicator solution while stirring.

**CAUTION:** Do not use your mouth to pipette the barium chloride solution. IT IS EXTREMELY POISONOUS.

e. Immediately titrate the mixture with the standard 0.02 normal hydrochloric acid to the first disappearance of the pink color (or to a pH of 8.3 with a pH meter). The color may reappear after a short time: do not continue the titration.

f. Report the alternate alkalinity, P1, as the cm<sup>3</sup> of 0.02 normal acid to reach the phenolphthalein endpoint.

g. Determine the blank alkalinity, P2. Omit the filtrate, but otherwise repeat the procedure described in Par. 8.10b through 8.10f for determining P1, using exactly the same quantities of water and reagents in preparing the sample.

h. Report the blank alkalinity, P2, as the cm<sup>3</sup> of 0.02 normal acid needed to titrate the reagent mixture to the phenolphthalein endpoint.

#### 8.11 Calculation — P1/P2 Alkalinity Method

a. The procedure given in the previous paragraphs is intended to reduce the major interferences in the P<sub>f</sub>/M<sub>f</sub> alkalinity test, and thus provide a better estimate of hydroxyl, carbonate, and bicarbonate concentrations. Calculating these concentrations does not make them true values; the composition is theoretical, based on water chemistry carbonate equilibria.

b. Within the limitations outlined the various ionic concentrations can be calculated as follows:

When P1 > P2

$$\text{OH}^-, \text{mg/L} = 340 (P_1 - P_2) \quad (a)$$

$$\text{CO}_3^{2-}, \text{mg/L} = 1200 [P_1 - (P_1 - P_2)] \quad (b)$$

When P1 < P2

$$\text{HCO}_3^-, \text{mg/L} = 1220 (P_2 - P_1) \quad (c)$$

$$\text{CO}_3^{2-}, \text{mg/L} = 1200 P_1 \quad (d)$$

#### CHLORIDE

8.12 Description. The chloride test measures the chloride-ion concentration in the mud filtrate.

#### 8.13 Equipment

a. Silver nitrate solution: containing 4.7910 g/L (equivalent to 0.001 g chloride-ion/cm<sup>3</sup>), stored in an amber or opaque bottle (CAS #7761-88-8)

b. Potassium chromate indicator solution: 5 g/100 cm<sup>3</sup> of water (CAS #7789-00-6)

c. Sulfuric or nitric acid solution: standardized 0.02 normal (N/50) (Sulfuric acid CAS #7664-93-9 or nitric acid CAS #7697-37-2)

d. Phenolphthalein indicator solution: 1 g/100 cm<sup>3</sup> of 50% alcohol/water solution (CAS #518-51-4)

e. Calcium carbonate: precipitated, chemically pure grade (CAS #471-34-1)

f. Distilled water

g. Serological (graduated) pipettes (TD): one 1-cm<sup>3</sup> and one 10-cm<sup>3</sup>

h. Titration vessel: 100-150-cm<sup>3</sup>, preferably white

i. Stirring rod

#### 8.14 Procedure

a. Measure one or more cm<sup>3</sup> of filtrate into the titration vessel. Add 2-3 drops of phenolphthalein solution. If

the indicator turns pink, add acid drop by drop from pipette, while stirring, until the color is discharged. If the filtrate is deeply colored, add an additional 2 cm<sup>3</sup> of 0.02 normal (N/50) sulfuric or nitric acid and stir. Then add 1 g of calcium carbonate and stir.

b. Add 25-50 cm<sup>3</sup> of distilled water and 5-10 drops of potassium chromate solution. Stir continuously, while adding standard silver nitrate solution drop by drop from the pipette, until the color changes from yellow to orange-red and persists for 30 seconds. Record the number of cm<sup>3</sup> of silver nitrate solution required to reach the endpoint. If over 10 cm<sup>3</sup> of silver nitrate solution are used, repeat the test with a smaller sample of filtrate.

**NOTE:** If the chloride-ion concentration of the filtrate exceeds 10,000 mg/L, a silver nitrate solution equivalent to 0.01 g chloride-ion per cm<sup>3</sup> may be used. The factor 1,000 in Equation 8.15 (a) is then changed to 10,000.

8.15 Calculation Report the chloride-ion concentration of the filtrate in mg/L, calculated as follows:

$$\text{Chloride, mg/L} = \frac{1000 (\text{silver nitrate, cm}^3)}{\text{filtrate sample, cm}^3} \quad (a)$$

To convert units:

$$\text{Chloride, ppm} = \frac{\text{Chloride, mg/L}}{\text{Specific Gravity of Filtrate}} \quad (b)$$

$$\text{Salt (NaCl), mg/L} = (1.65) (\text{Chloride, mg/L}) \quad (c)$$

Refer to Table 4.1 for Filtrate Specific Gravity.

Refer to Table 8.3 for conversion of % salt in salt water to mg/L or ppm.

#### TOTAL HARDNESS AS CALCIUM

8.16 Description The hardness of water or mud filtrate is due primarily to the presence of calcium and magnesium ions. When EDTA (ethylenediaminetetraacetic acid or its salt) is added to the water, it combines both with the calcium and magnesium and the endpoint is determined with a suitable indicator. The total hardness of the water is expressed as mg/L calcium. An endpoint obscured by dark components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

#### 8.17 Equipment

a. EDTA solution (Versenate): 0.01 molar: disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cm<sup>3</sup> = 1000 mg/L CaCO<sub>3</sub>, 1 cm<sup>3</sup> = 400 mg/L Ca<sup>2+</sup>) (CAS #139-33-3)

b. Buffer solution: 67.5 g ammonium chloride (CAS #12125-02-9) and 570 cm<sup>3</sup> ammonium hydroxide (CAS #1336-21-6) (15N) diluted to 1000 cm<sup>3</sup> with distilled water.

c. Hardness indicator solution: 1 g/L "Calmagite" or equivalent, 1-(1-hydroxy-4-methyl-2-phenylazo)-2-naphthol-4-sulfonic acid in distilled water (CAS #3147-14-6)

TABLE 8.3  
CONVERSION OF MILLIGRAMS PER LITER  
CHLORIDE TO WEIGHT PERCENT SALT (NaCl)  
OR PARTS PER MILLION SALT  
SOLUTION TEMPERATURE AT 68°F (20°C)

Milligrams Per Liter Chloride	Weight Percent Salt	Milligrams Per Liter Salt	Parts Per Million Salt
3.040	0.5	5,020	5,000
6.100	1	10,050	10,000
12.300	2	20,250	20,000
18.600	3	30,700	30,000
24.900	4	41,100	40,000
31.599	5	52,000	50,000
37.900	6	62,500	60,000
44.200	7	73,000	70,000
51.200	8	84,500	80,000
57.600	9	95,000	90,000
64.900	10	107,100	100,000
71.800	11	118,500	110,000
79.000	12	130,300	120,000
86,100	13	142,000	130,000
93,400	14	154,100	140,000
100,900	15	166,500	150,000
108,200	16	178,600	160,000
115,800	17	191,000	170,000
123,500	18	203,700	180,000
131,200	19	216,500	190,000
139,200	20	229,600	200,000
147,300	21	256,100	210,000
155,200	22	256,100	220,000
163,600	23	270,000	230,000
169,400	24	279,500	240,000
171,700	25	283,300	250,000
188,700	26	311,300	260,000

NOTE: It is quite common for many laboratories analyzing water samples to report milligrams per liter of salt as parts per million. If it is known that an analysis is reported in this way, the milligram per liter salt column should be used in converting to percent salt rather than the parts per million salt column.

d. Acetic acid: glacial (CAS #64-19-7) (CAUTION: Avoid skin contact).

e. Titration vessel: 150-cm<sup>3</sup> beaker

f. Serological (graduated) pipettes (TD): one 5-cm<sup>3</sup> and one 10-cm<sup>3</sup>

g. Volumetric pipettes (TD): one 1-cm<sup>3</sup>, one 2-cm<sup>3</sup>, and one 5-cm<sup>3</sup>

h. Hot plate (required if filtrate colored)

i. Masking agent: 1:1:2 mixture by volume of triethanolamine:tetraethylenepentamine:water (optional)

j. pH paper strip

k. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox®, or equivalent)

CAUTION: Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure the sodium hypochlorite is fresh as it will deteriorate with time.

1. Deionized or distilled water

NOTE: The deionized water and sodium hypochlorite solution should be tested for hardness by using 50.0 cm<sup>3</sup> of the deionized water in Par. 8.18f, and 10 cm<sup>3</sup> of the hypochlorite in Par. 8.18b, without the test sample, and continuing with Par. 8.18g and 8.18h. If the procedure is then repeated with the test sample utilizing 50.0 cm<sup>3</sup> of the deionized water and 10 cm<sup>3</sup> hypochlorite in Par. 8.18f, 8.18g, and 8.18h, the hardness of the test sample can be determined by subtracting the hardness of the deionized water and hypochlorite.

#### 8.18 Procedure

a. Measure 1.0 cm<sup>3</sup> or more of sample into a 150-cm<sup>3</sup> beaker. (If filtrate is clear, or is only lightly colored, omit steps 8.18b through 8.18e).

b. Add 10 cm<sup>3</sup> hypochlorite (Clorox® or equivalent) and mix.

c. Add 1 cm<sup>3</sup> glacial acetic acid and mix.

d. Boil the sample for five minutes. Maintain the sample volume by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required.

CAUTION: Work in an adequately ventilated area.

e. Cool the sample and wash the sides of the beaker with deionized water.

f. Dilute the sample to 50 cm<sup>3</sup> with deionized water. Add about 2 cm<sup>3</sup> of hardness buffer and swirl to mix.

NOTE: The presence of soluble iron may interfere with the endpoint determination. Should this be suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) has proven to be a suitable masking agent. One cm<sup>3</sup> of the mixture is used per titration.

g. Add sufficient hardness indicator (2-6 drops) and mix. A wine-red color will develop if calcium and/or magnesium is present.

h. While stirring, titrate with EDTA solution to the proper endpoint. Calcium indicators will produce a red to blue change. The endpoint is best described as that point where additional EDTA produces no further red to blue color change. The EDTA volume will be used in the calculation in Par. 8.19.

#### 8.19 Calculation

$$\text{Total hardness as calcium, mg/L} = \frac{400 (\text{EDTA volume, cm}^3)}{\text{volume sample, cm}^3} \quad (a)$$

## APPENDIX A

### CHEMICAL ANALYSIS OF WATER-BASED DRILLING FLUIDS

#### CALCIUM

**A.1 Description.** When EDTA (ethylenediaminetetraacetic acid or its salt) is added to water or mud filtrate containing both calcium and magnesium, it combines first with calcium. Calcium can be determined with EDTA when the pH of the sample is sufficiently high, so that magnesium is precipitated as the hydroxide, and an indicator specific for calcium is used. Several indicators will give color changes when all of the calcium has been complexed by EDTA at a pH of 12-13. An end point obscured by dark organic components can be remedied by oxidizing with a reagent such as sodium hypochlorite.

#### A.2 Equipment

a. EDTA solution (Versenate) (CAS #139-33-3): 0.01 molar, EDTA: disodium (ethylenediamine) tetraacetate dihydrate standardized (1 cm<sup>3</sup> = 1000 mg/L CaCO<sub>3</sub>, 1 cm<sup>3</sup> = 400 mg/L calcium).

b. Calcium buffer solution: 1N sodium hydroxide (NaOH) (CAS #1310-73-2)

c. Calcium indicator: Calver® II or hydroxy naphthol blue (CAS #63451-35-4)

d. Acetic acid: glacial (CAS #64-19-7)

**CAUTION:** Avoid skin contact.

e. Titration vessel: 150-cm<sup>3</sup> beaker

f. Serological (graduated) pipettes (TD): two 10-cm<sup>3</sup> and one 1-cm<sup>3</sup>

g. Volumetric pipettes (TD): one 1-cm<sup>3</sup>, one 2-cm<sup>3</sup>, and one 5-cm<sup>3</sup>

h. Hot plate (required if filtrate colored)

i. Masking agent: 1:1:2 mixture by volume of triethanolamine: tetraethylenepentamine: deionized water (optional).

j. pH paper

k. Graduated cylinder (TC): 50-cm<sup>3</sup>

l. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox® or equivalent)

**CAUTION:** Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure sodium hypochlorite is fresh as it will deteriorate with time.

m. Deionized or distilled water

**NOTE:** The deionized water and sodium hypochlorite solution should be tested for calcium by using 50.0 cm<sup>3</sup> of the deionized water in Par. A.3f and 10 cm<sup>3</sup> of the hypochlorite in Par. A.3b, without the test sample, and continuing with Par. A.3g and A.3h. If the procedure is

then repeated with the test sample utilizing 50.0 cm<sup>3</sup> of the deionized water and 10 cm<sup>3</sup> of the hypochlorite in Par. A.3f, A.3g, and A.3h, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and hypochlorite.

#### A.3 Procedure

a. With a volumetric pipette, add 1.0 cm<sup>3</sup> or more of sample to a 150-cm<sup>3</sup> beaker. This sample volume will be used in the calculation shown in A.4. If filtrate is colorless or is only slightly colored, omit steps A.3b through A.3e.

b. With serological pipette, add 10 cm<sup>3</sup> hypochlorite solution and mix.

c. With serological pipette, add 1 cm<sup>3</sup> glacial acetic acid and mix.

d. Boil the sample for five minutes. Maintain the sample by adding deionized water as required during boiling. Boiling is required to remove excess chlorine. The absence of chlorine can be verified by immersing a strip of pH paper in the sample. If the paper is bleached white, continued boiling is required. A sufficiently boiled sample will show a pH of 5.0.

**CAUTION:** Work in an adequately ventilated area.

e. Cool the sample and wash the sides of the beaker with deionized water.

f. Dilute the sample to approximately 50 cm<sup>3</sup> with deionized water. Add 10-15 cm<sup>3</sup> of NaOH buffer solution, or sufficient NaOH to produce a pH of 12-13.

**NOTE:** The presence of soluble iron may interfere with the endpoint determination. Should this be suspected, a mixture of triethanolamine:tetraethylenepentamine:water (1:1:2 by volume, respectively) is a suitable masking agent. Add 1.0 cm<sup>3</sup> of the mixture after A.3f.

g. Add sufficient calcium indicator (0.1-0.2 g) to produce a pink to wine-red color if calcium is present. Too much indicator will obscure the endpoint.

**NOTE:** The addition of several drops of methyl orange along with the calcium indicator may improve the visibility of the endpoint.

h. While stirring, titrate with standard EDTA to the proper endpoint. Calcium indicators will produce a red to blue change. The endpoint is best described as that point where additional EDTA produces no further red to blue color change. The EDTA volume will be used in the calculation in Par. A.4.

#### A.4 Calculation

$$\text{Calcium, mg/L} = \frac{400 (\text{EDTA volume, cm}^3)}{\text{volume sample, cm}^3} \quad (a)$$

## MAGNESIUM

**A.5 Description** The magnesium content of the mud filtrate can be calculated by subtracting the calcium ion content from the total hardness. This gives the magnesium content in terms of calcium which is converted to magnesium by multiplying the value by the ratio of atomic weights ( $24.3/40 = 0.6$ ).

### A.6 Procedure

- a. Determine the total hardness as calcium (Par. 8.18 through 8.21).
- b. Determine the calcium content (Appendix A.1 through A.4).

### A.7 Calculation

$$\text{Magnesium, mg/L} = (0.6)(\text{Total Hardness, mg/L} - \text{Calcium, mg/L})$$

## CALCIUM SULFATE

**A.8 Description** The calcium sulfate content of mud is determined by using the EDTA method as described in Par. A.1 through A.4 to determine the total calcium in a mud filtrate and the whole mud. The total and undissolved calcium sulfate contents of the mud can then be calculated.

### A.9 Equipment

- a. EDTA solution (Versenate): 0.01 molar EDTA: disodium (ethylenediamine)tetraacetate dihydrate standardized, ( $1 \text{ cm}^3 = 1000 \text{ mg/L CaCO}_3$ ,  $1 \text{ cm}^3 = 400 \text{ mg/L Ca}^{+2}$ ) (CAS #139-33-3)
  - b. Buffer solution: 1N sodium hydroxide (NaOH) (CAS #1310-73-2)
  - c. Calcium indicator: Calver® II or equivalent, or hydroxy naphthol blue (CAS #63451-35-44)
  - d. Acetic acid: glacial (CAS #64-19-7)
- CAUTION:** Avoid skin contact
- e. Titration vessel: 150-cm<sup>3</sup> beaker
  - f. Serological (graduated) pipettes (TD): two 10-cm<sup>3</sup> and one 1-cm<sup>3</sup>
  - g. Volumetric pipettes (TD): one 1-cm<sup>3</sup>, one 2-cm<sup>3</sup>, one 5-cm<sup>3</sup>, one 10-cm<sup>3</sup>
  - h. Hot plate (required, if filtrate colored)
  - i. Masking agent: 1:1:2 mixture by volume of triethanolamine:tetraethylenepentamine:deionized water (optional)
  - j. pH paper
  - k. Graduated cylinder (TC): 50-cm<sup>3</sup>

l. Mud retort, as described in RP 13B-1, Water-Based Drilling Fluids, Section 4.

m. Sodium hypochlorite solution: 5.25% sodium hypochlorite in deionized water (i.e., Clorox® or equivalent)

**CAUTION:** Many brands contain calcium hypochlorite or oxalic acid and should not be used. Be sure sodium hypochlorite is fresh as it will deteriorate with time.

n. Deionized or distilled water

**NOTE:** The deionized water and sodium hypochlorite solution should be tested for calcium by using 10.0 cm<sup>3</sup> of the deionized water in Par. A.10a and 10 cm<sup>3</sup> of the hypochlorite in Par. A.10a, without the test sample, and continuing with Par. A.10a through A.10c. If the procedure is then repeated with the test sample utilizing 10.0 cm<sup>3</sup> of the deionized water and 10 cm<sup>3</sup> of the sodium hypochlorite in Par. A.10a, A.10b, and A.10c, the calcium of the test sample can be determined by subtracting the calcium of the deionized water and sodium hypochlorite.

### A.10 Procedure

a. Add 5 cm<sup>3</sup> of whole mud to 245 cm<sup>3</sup> of deionized water. Stir the mixture for 15 minutes and filter through a standard API filter press. Collect only clear filtrate. Into a 150-cm<sup>3</sup> beaker, add 10 cm<sup>3</sup> of clear filtrate with the 10-cm<sup>3</sup> volumetric pipette and titrate to the EDTA endpoint as described in Par. A.1 through A.4. Call this volume of EDTA  $V_t$ .

b. Titrate 1 cm<sup>3</sup> of the original mud filtrate (obtained as described in Paragraph 3.1 through 3.3) to the EDTA endpoint. Call this volume of EDTA  $V_f$ .

c. Retort the mud. Determine the volume fraction of water in the mud,  $F_w$ , by using the value for volume percent water from the liquid and solids determination and the following equation:

$$F_w = \frac{\text{Volume percent water}}{100} \quad (a)$$

### A.11 Calculation

a. The calcium sulfate content of the mud in lb/bbl is calculated by using the following equation:

$$\text{Total calcium sulfate, lb/bbl} = 2.38 (V_t) \quad (a)$$

b. The (excess) undissolved calcium sulfate content of the mud in lb/bbl may be calculated by using the subsequent equation:

$$\text{Excess calcium sulfate, lb/bbl} = 2.38 (V_t) - 0.48 (V_f F_w) \quad (b)$$

## FORMALDEHYDE

**A.12 Description** This procedure is for the determination of paraformaldehyde content of drilling fluid. Sodium sulfite is reacted with a filtrate sample (neutralized to a phenolphthalein endpoint) which is then titrated with the acid to the phenolphthalein endpoint again. A blank must be run to subtract the contribution to alkalinity attributable to sodium sulfite. The difference in two titrations is the amount of paraformaldehyde present in the drilling fluid.

## A.13 Equipment

a. Phenolphthalein indicator: 1 g/100 cm<sup>3</sup> of 50% alcohol/water (CAS #518-51-4)

b. Sodium hydroxide solution: 0.02 normal (N/50) (CAS #1310-73-2)

c. Sulfuric acid solution: 0.02 normal (N/50) (CAS #7664-93-9)

d. Sodium sulfite solution: 4 g/100 cm<sup>3</sup> of distilled water (CAS #7757-83-7)

NOTE: This solution deteriorates rapidly. If older than 30 days, it should be replaced with a fresh solution.

e. Titration vessel: a test tube or a casserole

f. Serological (graduated) pipette (TD): 10-cm<sup>3</sup>

g. Volumetric pipettes (TD): one 1-cm<sup>3</sup> and one 3-cm<sup>3</sup>

## A.14 Procedure

a. Pipette 3 cm<sup>3</sup> of the mud filtrate into the casserole or test tube. Add 2 drops of phenolphthalein indicator solution. If the sample remains colorless, add sodium hydroxide solution drop by drop, with agitation, until a faint pink color develops. Then add sulfuric acid drop by drop to dispel the color.

b. If, upon the first addition of phenolphthalein, the filtrate becomes colored, add sulfuric acid dropwise until the color is just dispelled.

c. To the neutralized filtrate, add 1 cm<sup>3</sup> of sodium sulfite solution; a red color will develop.

d. After approximately 30 seconds, titrate with sulfuric acid until the sample is a very faint pink. Record the amount of acid used in cm<sup>3</sup>. This is the amount of sulfuric acid which will be used for the calculation, and noted as  $V_f$ .

e. Repeat the foregoing Pars. A.14c through A.14d using distilled water instead of mud filtrate. Record the amount of acid used in cm<sup>3</sup>. This will be used for the calculation in A.15, and noted as  $V_b$ .

## A.15 Calculation

$$\text{Formaldehyde content,} = (0.07) (V_f - V_b) \quad (a) \\ \text{lb/bbl}$$

## SULFIDE

## A.16 Description

a. The concentration of soluble sulfides in a drilling fluid can be determined by this method. Soluble sulfides include H<sub>2</sub>S and the sulfide (S<sup>2-</sup>) and bisulfide (HS<sup>-</sup>) ions. Mud filtrate is acidified in a Garrett Gas Train, converting all sulfides to H<sub>2</sub>S which is evolved by bubbling an inert carrier gas through the sample. The Gas Train separates the gas from the liquid. The gas stream is passed through a Dräger tube which responds to H<sub>2</sub>S by darkening along its length. The

darkened length is proportional to the total sulfide in the mud filtrate. The low range Dräger tube turns from white to brownish-black and the high range Dräger tube turns from pale blue to jet-black. No common mud contaminant will cause these color changes.

b. Lead-acetate paper disks can be accommodated in the Garrett Gas Train to determine the presence or absence of sulfide. If the presence of sulfide is indicated by darkening of the lead-acetate paper, a Dräger tube should be used for quantitative analysis.

## A.17 Equipment

a. The Garrett Gas Train apparatus, as shown in Fig. A.1, consists of: a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter and a Dräger tube. Specifications of Garrett Gas Train:

## Body

## Chamber 1

Depth ..... 3.54 in. (90 mm)  
Diameter ..... 1.52 in. (38 mm)

## Chambers 2 &amp; 3

Depth ..... 3.54 in. (90 mm)  
Diameter ..... 1.18 in. (30 mm)

## Passages between Chambers:

Diameter ..... 0.08 in. (2.0 mm)

## Material:

Lucite® or equivalent transparent material or glass which is inert to acid, sulfides, and hydrogen sulfide gas.

## Dispersion Tube

## Stem:

Diameter ..... 0.315 in. (8.0 mm)  
Length ..... approx. 5.9 in. (150 mm)

## Dispersion Frit: (Bell-shaped ASTM 4-5.5 Fine)

Diameter ..... 1.18 in. (30 mm)  
Pyrex® or equivalent glass

Flow Meter: Floating ball type preferred, capable of measuring 300 cm<sup>3</sup> per minute of CO<sub>2</sub> gas.

Flexible Tubing: Type inert to hydrogen sulfide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and Rigid Tubing: Type inert to hydrogen sulfide and acid.

b. Carrier gas: type inert to hydrogen sulfide, acid, and Dräger tube reagents. Nitrogen is preferred but carbon dioxide is acceptable. (Avoid air or other oxygen-containing gases.)

c. Dräger H<sub>2</sub>S Analysis Tubes:

(1) Low range: marked H<sub>2</sub>S 100/a (No. CH-291-01)

(2) High range: marked H<sub>2</sub>S 0.2%/A (No. CH-281-01)

d. Lead-acetate paper disk (see Note Par. A.18o)

e. Sulfuric acid (CAS #7664-93-9): approximately 5N, reagent grade



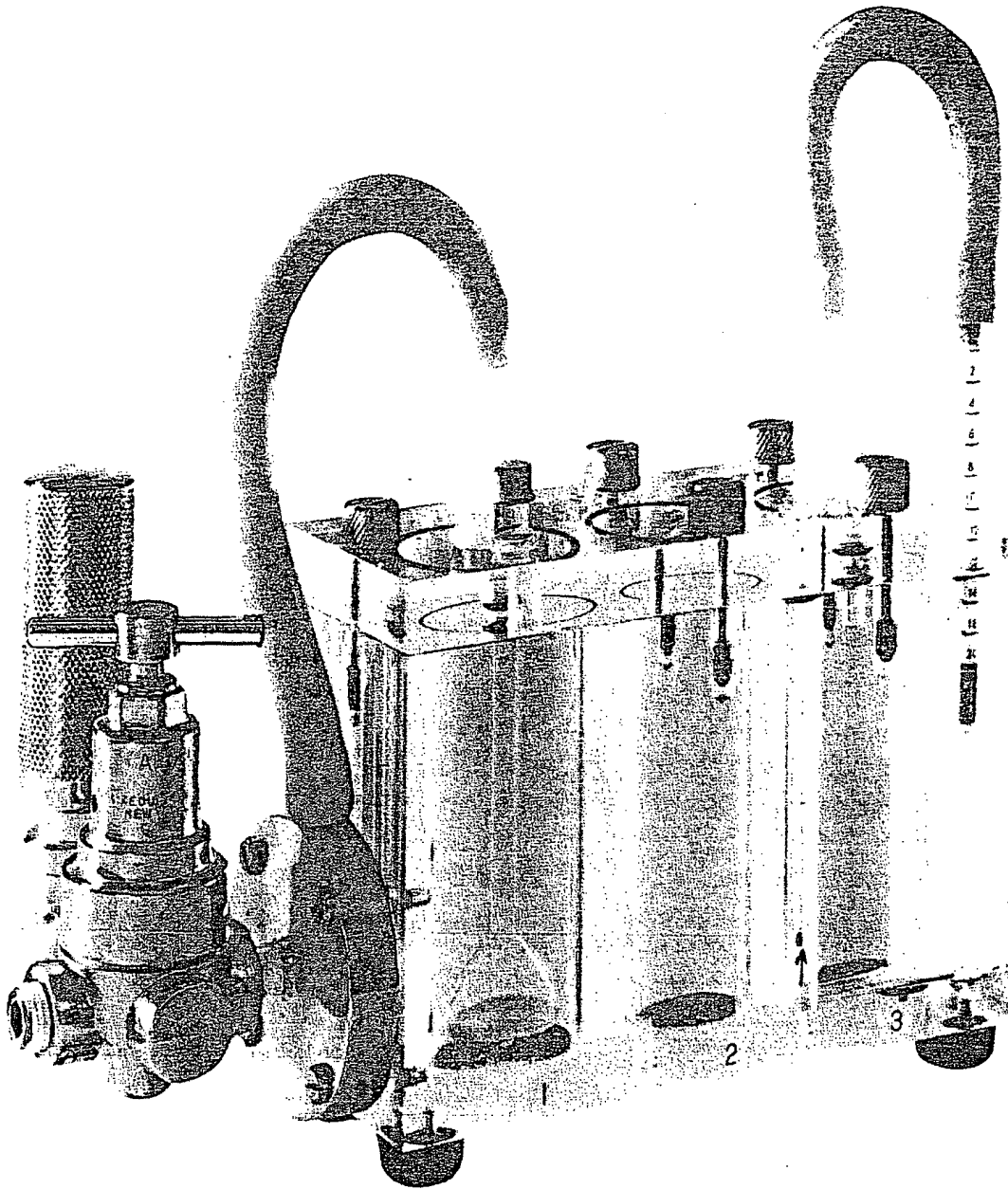


FIG. A.1  
ANALYSIS OF SOLUBLE SULFIDES



- f. Defoamer in a dropper bottle
- g. Hypodermic syringes: one 10-cm<sup>3</sup> (for acid), and one 2.5-cm<sup>3</sup>, one 5-cm<sup>3</sup> and one 10-cm<sup>3</sup> (for sample)
- h. Hypodermic needles: two 1.5 in. (38 mm) 21-gauge needles

#### A.18 Procedure

- a. Be sure the gas train is clean, dry and on a level surface, with the top removed.

*NOTE: Moisture in the train can cause the ball in the flowmeter to float erratically and may affect the accuracy of the Dräger tube reading.*

- b. Add 20 cm<sup>3</sup> of deionized water to Chamber 1.

- c. Add 5 drops defoamer to Chamber 1.

- d. See Table A.1 for sample volume and type of Dräger tube required for the expected sulfide range. Select the proper type Dräger tube. Break the tip from each end of the tube.

- e. Install the Dräger tube with the arrow pointing downward into the bored receptacle. Likewise, install the flowmeter tube with the word TOP upward. Be sure o-rings seal around the body of each tube.

**TABLE A.1**  
**DRÄGER TUBE IDENTIFICATION, SAMPLE**  
**VOLUMES AND TUBE FACTORS TO BE USED**  
**FOR VARIOUS SULFIDE RANGES**

Sulfide Range (mg/L)	Sample Volume (cm <sup>3</sup> )	Dräger Tube I.D. (See Tube Body)	Tube Factor (See A.19a)
1.2 - 24	10.0	H <sub>2</sub> S 100/a	12
1.5 - 48	5.0		
4.8 - 96	2.5		
60 - 1020	10.0		
120 - 2040	5.0	H <sub>2</sub> S 0.2%/A	600*
240 - 4080	2.5		

\*"Tube Factor" 600 is based on a "Batch Factor" (stenciled on box) of 0.40. For another "Batch Factor" (as stenciled on box), a corrected "Tube Factor" should be calculated:

$$\text{Correct Tube Factor} = \frac{600 (\text{Batch Factor})}{0.40} \quad (a)$$

- f. Install the top on the gas train and hand tighten all screws evenly to seal the o-rings.

- g. With the regulator backed off, connect the carrier gas to the dispersion tube of Chamber 1 using flexible tubing. If CO<sub>2</sub> cartridge is used, install and puncture cartridge and connect to dispersion tube as shown in Fig. A.1.

- h. Attach the flexible tubing from Chamber 3 outlet to the Dräger tube, as shown in Fig. A.1.

*NOTE: Use only latex rubber or inert plastic tubing. Do not clamp flexible tubing; unclamped tubing provides pressure relief in the event of over pressurization.*

- i. Adjust the dispersion tube in Chamber 1 to approximately ¼ in. (5 mm) above the bottom.

- j. Gently flow carrier gas for a 30 second period to purge air from the system. Check for leaks. Shut off the carrier gas.

- k. Collect a sufficient volume of solids-free filtrate for analysis. (If a low concentration of soluble sulfides is to be detected, a large volume of filtrate is required. Use Table A.1 as a guide.)

- l. Inject a measured volume of the solids-free filtrate sample into Chamber 1 through the rubber septum, using a hypodermic syringe and needle.

- m. Slowly inject 10 cm<sup>3</sup> sulfuric acid solution into Chamber 1 through rubber septum using the hypodermic syringe and needle.

- n. Immediately restart the carrier gas flow. The flow rate should be maintained between 200-400 cm<sup>3</sup> per minute.

*NOTE: One CO<sub>2</sub> cartridge should provide about 15-20 minutes of flow at this rate.*

- o. Observe changes in appearance of the Dräger tube. Note and record the maximum darkened length (in units marked on the tube) before the front starts to smear. Continue flowing for a total of 15 minutes although the front may attain a diffuse and feathery coloration. In the high-range tube an orange color (caused by SO<sub>2</sub>) may appear ahead of the black front if sulfites are present in the sample. The orange SO<sub>2</sub> region should be ignored when recording darkened length.

*NOTE: For best Dräger tube accuracy, the "Darkened Length" should fill more than half the tube's length, therefore the filtrate "Sample Volume" must be carefully selected.*

- p. A lead-acetate paper disk fitted under the o-ring of Chamber 3 can be substituted for the Dräger tube in the Gas Train. The lead-acetate paper will show qualitatively the presence or absence of sulfides in the sample. A dark discoloration of the paper is a positive indication of sulfides. After the positive indication, the Dräger tube should be used on a separate sample for quantitative analysis.

- q. To clean the gas train remove the flexible tubing and remove the top. Take Dräger tube and flow-meter out of the receptacles and plug the holes with stoppers to keep them dry. Wash out the chambers with warm water and mild detergent, using a soft brush. Use a pipe cleaner to clean the passages between the chambers. Wash, rinse and blow out the dispersion tube with a dry gas. Rinse the unit with deionized water and allow to drain dry.

## A.19 Calculation

a. Using the measured Sample Volume, the Dräger tube's maximum Darkened Length and the Tube Factor from Table A.1, calculate sulfide in the sample:

$$\text{Sulfide, mg/L} = \frac{(\text{Darkened Length}^*) (\text{Tube Factor})}{(\text{Sample Volume, cm}^3)} \quad (\text{a})$$

\*In units marked on the tube

## CARBONATE

A.20 Description The concentration of soluble carbonates in a drilling fluid filtrate can be determined by this method. Total soluble carbonates include  $\text{CO}_2$  and the carbonate ( $\text{CO}_3^{2-}$ ) and bicarbonate ( $\text{HCO}_3^-$ ) ions. Mud filtrate is acidified in a Garrett Gas Train, converting all carbonates to  $\text{CO}_2$ , which is then evolved by bubbling an inert carrier gas through the sample. The Gas Train separates the gas from the liquid. The gas stream is collected in a one liter gas bag (to allow  $\text{CO}_2$  to mix uniformly) and subsequently drawn through a Dräger tube at a fixed flow rate. The Dräger tube responds to  $\text{CO}_2$  by progressively staining purple along its length. A reaction between  $\text{CO}_2$  and a hydrazine chemical causes a crystal violet indicator to turn purple. The stain length is proportional to the total carbonate concentration in the filtrate.

## A.21 Equipment

a. Garrett Gas Train apparatus, as shown in Fig. A.2, consists of: a transparent plastic gas train, an inert gas supply and pressure regulator, a floating-ball flowmeter and a Dräger tube. Specifications of Garrett Gas Train:

## Body

## Chamber 1

Depth ..... 3.54 in. (90 mm)  
Diameter ..... 1.52 in. (38 mm)

## Chambers 2 &amp; 3

Depth ..... 3.54 in. (90 mm)  
Diameter ..... 1.18 in. (30 mm)

## Passages between Chambers:

Diameter ..... 0.08 in. (2.0 mm)

## Material:

Lucite® or equivalent transparent material or glass which is inert to acid, carbonates, and carbon dioxide gas.

## Dispersion Tube

## Stem:

Diameter ..... 0.315 in. (8.0 mm)  
Length ..... approximately 5.9 in. (150 mm)

## Dispersion Frit: (bell-shaped ASTM 4-5.5 Fine)

Diameter ..... 1.18 in. (30 mm)  
Pyrex® or equivalent glass

Flexible Tubing: Type inert to carbon dioxide and carrier gas. Latex rubber or Tygon® plastic or equivalent is preferred.

Fittings and Rigid Tubing: type inert to carbon dioxide and acid.

b. Carrier gas: nitrogen ( $\text{N}_2$ ) bottle with low-pressure regulator (preferred), or  $\text{N}_2\text{O}$  gas cartridges (e.g., WHIPPETS #561241 by Walter Kidde and Co. Inc., Belleville, N.J.) or equivalent.

**CAUTION:** Nitrous oxide cartridges should not be used as pressure sources for high temperature, high pressure (HTHP) filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

c. Dräger  $\text{CO}_2\text{O}$  analysis tube: marked  $\text{CO}_2$  0.01%/a (No. CH-308-01).

d. Dräger one-liter ALCOTEST gas bag No. 7626425, or equivalent.

e. Dräger MULTIGAS DETECTOR hand-operated vacuum pump, Model 31, or equivalent.

f. Stopcock (2-way bore), 8 mm glass with Teflon® plug.

g. Sulfuric Acid (CAS #7664-93-9): approximately 5N, reagent grade.

h. Defoamer in a dropper bottle.

i. Hypodermic syringes: one 10-cm<sup>3</sup> (for acid) and one 1.0-cm<sup>3</sup>, one 5-cm<sup>3</sup> and one 10-cm<sup>3</sup> (for sample).

j. Hypodermic needles: two 1.5-inch (38 mm) 21-gauge needles.

**NOTE:** Nitrogen is preferred over  $\text{N}_2\text{O}$  as the carrier gas. Because  $\text{N}_2\text{O}$  cools upon expansion and chills the diaphragm in the regulator, prolonged  $\text{N}_2\text{O}$  flow will cause the regulator to perform erratically.

## A.22 Procedure

a. Be sure that the gas train is clean, dry and on a level surface, with the top removed.

**NOTE:** If  $\text{CO}_2$  has been used as the carrier gas in the previous test (i.e., sulfide analysis), the regulator, tubing and dispersion tube should be purged with carrier gas at this time.

b. Add 20 cm<sup>3</sup> deionized water to Chamber 1.

c. Add 5 drops of defoamer to Chamber 1.

d. Install the top on the gas train and hand-tighten evenly to seal all o-rings.

e. Adjust the dispersion tube to approximately ¼ in. (5 mm) off bottom.

f. With regulator backed off, connect carrier gas supply to glass dispersion tube of Chamber 1 using flexible tubing.

g. Flow carrier gas through train for one minute to purge air from the system. Check for leaks in gas train unit.

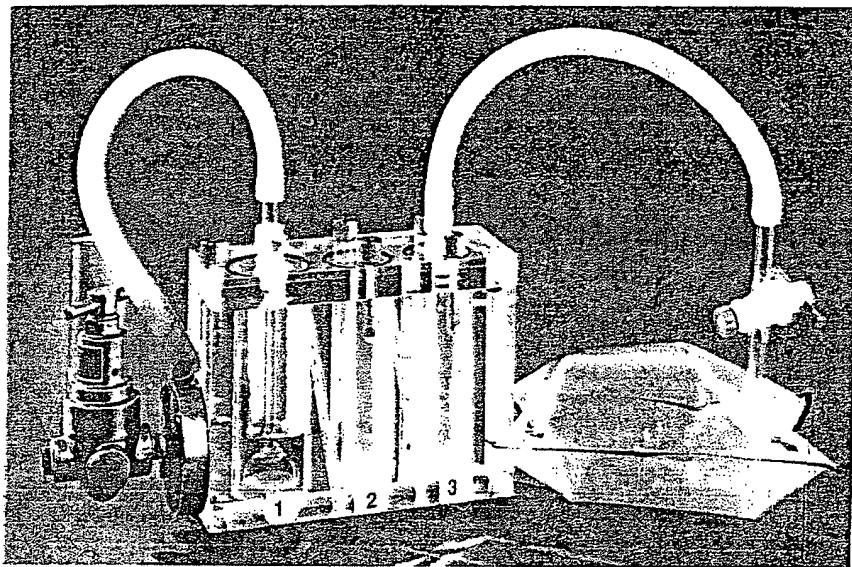


FIG. A.2  
Step 1 — Freeing Carbonates as  $\text{CO}_2$  Gas

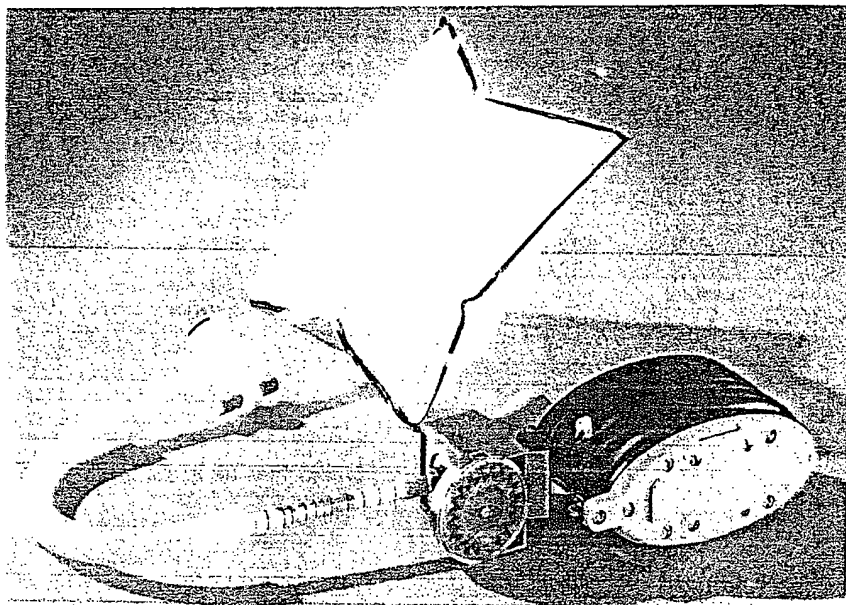


FIG. A.3  
Step 2 — Analyzing  $\text{CO}_2$  with Dräger Tube  
ANALYSIS OF SOLUBLE CARBONATE

h. Fully collapse the gas bag and simultaneously check the system for leaks. To do this, connect the gas bag and stopcock to hand pump, as shown in Fig. A.3. (Use a discarded Dräger tube as connection and start with the bag essentially empty.) Fully depress and release the hand pump. When the bag is completely empty and free of leaks, the pump will remain depressed for several minutes. If leakage is detected, check the pump and all connections. To check the pump alone, insert a sealed Dräger tube into the pump opening and depress bellows. It will remain depressed if pump does not leak.

i. With the bag fully collapsed, install flexible tubing from the stopcock and bag onto the outlet of Chamber 3, as seen in Fig. A.2.

j. Inject a measured volume of solids-free filtrate into Chamber 1 through septum with a hypodermic syringe and needle. See Table A.2.

TABLE A.2  
DRÄGER TUBE IDENTIFICATION, SAMPLE  
VOLUMES AND TUBE FACTOR TO BE USED  
FOR VARIOUS CARBONATE RANGES

Carbonate Range (mg/L)	Sample Volume (cm <sup>3</sup> )	Dräger Tube Identification (see Tube Body)	Tube Factor (see A.23a)
25 - 750	10.0		
50 - 1500	5.0	CO <sub>2</sub> 0.01%/a	25,000
250 - 7500	1.0		

k. Slowly inject 10 cm<sup>3</sup> sulfuric acid solution into Chamber 1 through rubber septum using a clean syringe and needle. Gently shake the gas train to mix acid with sample in Chamber 1.

l. Open the stopcock on the gas bag. Restart gas flow and allow gas bag to fill steadily during a 10 minute interval. When bag is firm to the touch (do not burst it) shut off flow and close the stopcock. Immediately proceed to next step.

m. Break the tip off each end of the Dräger tube.

n. Remove the tubing from Chamber 3 outlet and reinstall it onto the upstream end of the Dräger tube. (Observe that an arrow on the tube indicates gas flow direction.) Attach Dräger hand pump to downstream end of Dräger tube, as shown in Fig. A.3.

o. Open the stopcock on the bag. With steady hand-pressure fully depress the hand pump. Release pump so that gas flows out of the bag and through the Dräger tube. Operate the pump and count the strokes until the bag is empty. (Ten strokes should empty the bag. More than ten strokes indicates leakage has occurred and test results will not be correct.)

p. Observe a purple stain on the Dräger tube if CO<sub>2</sub> is present in the gas bag and record the stain length in units marked on the Dräger tube. (Include the faint blue tinge in the purple stain length reading.)

NOTE: For best Dräger tube accuracy, the "Stain Length" should fill more than half the tube length, therefore "Sample Volume" must be carefully selected.

q. To clean the gas train remove the flexible tubing and remove the top. Wash out the chambers with warm water and mild detergent, using a brush. Use a pipe cleaner to clean the passages between chambers. Wash, rinse and then blow out the dispersion tube with dry gas. Rinse the unit with deionized water and allow to drain dry. Be sure to periodically replace the disposable gas bag to avoid leaks and contamination in the bag. (Bag replacement is suggested after 10 analyses.)

#### A.23 Calculation

a. Using the measured "Sample Volume," the Dräger tube "Stain Length" and "Tube Factor" of 25,000 (see Table A.2), calculate total soluble carbonates (CO<sub>2</sub> + CO<sub>3</sub><sup>-2</sup> + HCO<sub>3</sub><sup>-</sup>) in the filtrate sample using equation:

$$\text{Carbonate, mg/L} = \frac{25,000 (\text{Stain Length})}{\text{Sample Volume, cm}^3} \quad (\text{a})$$

#### POTASSIUM ABOVE 5000 mg/L

A.24 Description. Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the properties of the drilling fluid. This procedure is used to measure the potassium ion content in mud filtrates at levels above 5000 mg/L or 3.5 lb/bbl KCl. Potassium is precipitated in a centrifuge tube as the perchlorate salt, and then the precipitate volume is measured. The potassium ion content is read from a prepared standard curve.

#### A.25 Equipment

a. Sodium perchlorate solution (CAS #7601-89-0): 150.0 g NaClO<sub>4</sub>/100 cm<sup>3</sup> distilled water.

NOTE: Sodium and potassium perchlorates are explosive in the dry state if heated or if in contact with organic reducing agents. The perchlorates are not hazardous if kept wet. They will decompose harmlessly in water.

b. Standard potassium chloride solution (CAS #7447-40-7): 14.0 g KCl made up to 100 cm<sup>3</sup> with deionized or distilled water.

c. Centrifuge, horizontal-swing rotor head (manual or electric) capable of producing approximately 1800 rpm (see Fig. A.4)

NOTE: A fairly constant 1800 rpm can be obtained with a manual centrifuge as follows:

1. Determine the number of revolutions of the rotor per each turn of the crank; i.e., move the crank very slowly and count the number of revolutions of the rotor head during one turn of the crank. For example, 15 revolutions of the rotor per one turn of the crank.

2. Calculate the number of crank turns required to obtain 1800 revolutions of the rotor head. In the example, to obtain 1800 revolutions of the head would require 120 turns of the crank (1800/15). Thus the crank must be turned 120 times in one minute to obtain the rate of 1800 rpm. At this rate, in 5 seconds the handle must be turned 10 times (120/60) (5). By counting the crank turns in 5 seconds and adjusting the rate to obtain the required number of turns, a constant 1800 rpm should be obtained in 15-20 seconds. The interval used to adjust to the 1800 rpm should be included in the centrifuge time of the sample.

d. Clinical centrifuge tube: 10-cm<sup>3</sup> Kolmer type (do not substitute), e.g., Corning #8360 (see Fig. A.5).

e. Volumetric pipettes (TD): one 0.5-cm<sup>3</sup>, one 1.5-cm<sup>3</sup>, one 2.5-cm<sup>3</sup>, and 3-cm<sup>3</sup>

f. Hypodermic syringe or serological (graduated) pipette (TD): 10-cm<sup>3</sup>

g. Distilled or deionized water

#### A.26 Procedure — Standard Calibration Curve

A standard calibration curve is required for each type of centrifuge. A minimum of three points (3.5, 10.5 and 17.5 lb/bbl KCl) is required to obtain an accurate graph.

a. Samples can be prepared by using the standard potassium chloride solution (0.5 cm<sup>3</sup> of standard potassium chloride solution is equivalent to 3.5 lb/bbl KCl). To obtain 3.5, 10.5, and 17.5 lb/bbl KCl, use 0.5, 1.5 and 2.5 cm<sup>3</sup> of the standard potassium chloride solution, respectively.

b. Dilute the sample to the 7.0 cm<sup>3</sup> mark with distilled water and agitate.

c. Add 3.0 cm<sup>3</sup> of standard sodium perchlorate solution: (DO NOT AGITATE.)

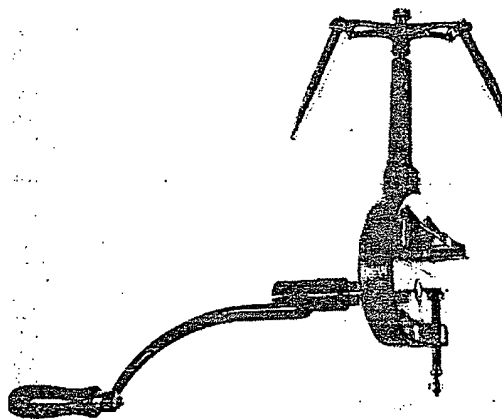


FIG. A.4  
MANUAL CENTRIFUGE  
WITH HORIZONTAL-SWING ROTOR HEAD

d. Centrifuge at a constant speed (approximately 1800 rpm) for one minute and read the precipitate volume immediately.

**CAUTION:** Counterbalance the centrifuge tube with another tube and liquid of the same weight.

e. Clean the centrifuge tube immediately after use to facilitate ease of cleaning.

f. Plot the volume of precipitate (cm<sup>3</sup>) versus potassium chloride content (lb/bbl) on rectangular graph paper as shown in Fig. A.6.

#### A.27 Procedure — Sample Testing

a. Measure the appropriate volume of filtrate into the centrifuge tube (see Table A.3 for range).

b. Dilute to 7.0 cm<sup>3</sup> if necessary with distilled water, and agitate.

c. Add 3.0 cm<sup>3</sup> of standard sodium perchlorate solution. (DO NOT AGITATE.) If potassium is present, precipitation occurs at once.

d. Centrifuge at constant speed (approximately 1800 rpm) for one minute. Read the precipitate volume immediately and record.

**NOTE:** Counterbalance the centrifuge tube with another tube and liquid of the same weight.

e. Add 2-3 drops of the sodium perchlorate solution to the tube. If precipitate still forms the total amount of potassium was not measured. (See Table A.3 and use the next smaller filtrate volume and repeat Par. A.27a through A.27e.)

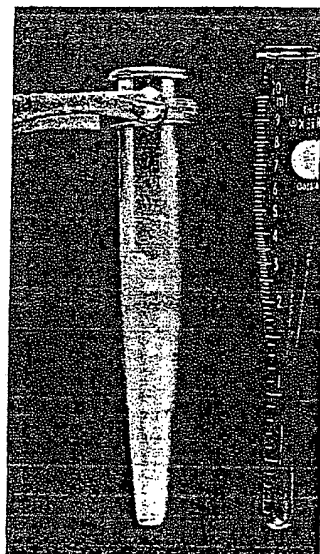


FIG. A.5  
10-CM<sup>3</sup> CLINICAL CENTRIFUGE TUBE  
KOLMER TYPE

f. Determine the potassium chloride concentration by comparing the precipitate volume measured with the standard calibration curve as prepared in Par. A.26. Report the potassium concentration as lb/bbl KCl ( $\text{kg}/\text{m}^3$ ). The potassium concentration may also be reported as mg/L potassium ion. If the filtrate potassium chloride concentration from the standard calibration curve exceeds an 18 lb/bbl reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in Table A.3, and repeat A.27a through A.27e.

#### A.28 Calculation

$$\text{KCl in filtrate, lb/bbl} = \frac{7}{\text{cm}^3 \text{ filtrate used}} \left( \text{value from standard curve} \right) \quad (\text{a})$$

$$\text{K}^+ \text{ in filtrate, mg/L} = 1500 (\text{KCl in filtrate, lb/bbl}) \quad (\text{b})$$

TABLE A.3  
FILTRATE VOLUMES TO BE USED AT  
VARIOUS KCl CONCENTRATIONS

Concentration Range KCl (lb/bbl)	K <sup>+</sup> (mg/L)	Filtrate Volume To Use (cm <sup>3</sup> )
3.5-18	5,250 - 27,000	7.0
18-35	27,000- 52,500	3.5
35-70	52,500-105,000	2.0
over 70	over 105,000	1.0

#### POTASSIUM BELOW 5000 mg/L

A.29 Description This procedure is used to measure potassium ion content in mud filtrates at levels below 5000 mg/L. Potassium ion is precipitated as the tetraphenylborate salt by adding an excess of standard sodium tetraphenylborate (STPB) solution. The unreacted STPB is then determined by titration with a quaternary ammonium salt (QAS), hexadecyltrimethyl ammonium bromide, using bromophenol blue as an indicator. The endpoint is a purple-blue to light blue color change. The potassium ion concentration in the sample is calculated by subtracting the amount of unreacted STPB from the amount of STPB originally added to the sample.

#### A.30 Equipment

a. Standard sodium tetraphenylborate solution (STPB) (CAS #143-66-8): 8.754 g in 800 cm<sup>3</sup> deionized water. Add 10-12 g aluminum hydroxide, stir 10 minutes and filter. Add 2 cm<sup>3</sup> of 20% NaOH solution to the filtrate and dilute to one liter with deionized water.

b. Quaternary ammonium salt solution (QAS) (CAS #57-09-0): 1.165 g hexadecyltrimethyl ammonium bromide/500 cm<sup>3</sup> deionized water

c. Sodium hydroxide solution (CAS #1310-73-2): 20 g/80 cm<sup>3</sup> deionized water

d. Bromophenol blue indicator (CAS #115-39-9): 0.04 g tetrabromophenolsulfonphthalein/3 cm<sup>3</sup> 0.1N NaOH. Dilute to 100 cm<sup>3</sup> with deionized water.

e. Deionized or distilled water

f. Serological (graduated) pipettes (TD): one 2-cm<sup>3</sup> graduated in 0.01 cm<sup>3</sup> subdivisions, two 5-cm<sup>3</sup>, and two 10-cm<sup>3</sup>

g. Graduated cylinders: two 25-cm<sup>3</sup> (TD) and two 100-cm<sup>3</sup> (TC)

h. Beakers: two 250-cm<sup>3</sup>

i. Funnel

j. Filter paper

#### A.31 Procedure

a. Place the proper amount of filtrate into a 100-cm<sup>3</sup> graduated cylinder, using Table A.4 to determine sample size. Be sure to use a pipette to measure the amount of filtrate.

TABLE A.4  
FILTRATE VOLUMES TO BE USED AT LOW  
KCl CONCENTRATIONS

Concentration Range KCl (lb/bbl)	K <sup>+</sup> (mg/L)	Filtrate Volume To Use (cm <sup>3</sup> )
0.2-1.3	250- 2,000	10.0
1.3-2.7	2,000- 4,000	5.0
2.7-6.7	4,000-10,000	2.0

b. Add 4 cm<sup>3</sup> of 20% NaOH solution (measured with a 5-cm<sup>3</sup> pipette), 25 cm<sup>3</sup> of STPB solution (measured with a 25-cm<sup>3</sup> graduated cylinder), and enough deionized water to bring the level of the solution to 100 cm<sup>3</sup> mark.

c. Mix and allow to stand ten minutes.

d. Filter into a 100-cm<sup>3</sup> graduated cylinder. If the filtrate is cloudy, the solution must be refiltered.

e. Transfer 25 cm<sup>3</sup> of the above filtrate (measured with a 25-cm<sup>3</sup> graduated cylinder) into a 250-cm<sup>3</sup> beaker.

f. Add 10-15 drops of bromophenol blue indicator.

g. Titrate with QAS solution until color changes from purple-blue to light blue.

NOTE: It is important to check the concentration of QAS solution versus the STPB solution at monthly intervals. To determine the equivalent QAS, dilute 2 cm<sup>3</sup> of the STPB solution in a titration vessel with 50 cm<sup>3</sup> deionized water. Add 1 cm<sup>3</sup> of 20% NaOH solution and 10-20 drops of the bromophenol blue indicator. Titrate with the QAS solution until color changes from purple-blue to light blue.

$$\text{Ratio of QAS to STPB} = \frac{\text{QAS, cm}^3}{2} \quad (\text{a})$$

If the ratio is other than  $4.0 \pm 0.5$ , calculate a correction factor to be used in the calculation of mg/L  $K^+$

$$\text{Correction factor (CF)} = \frac{8}{\text{QAS, cm}^3} \quad (b)$$

#### A.32 Calculation

$$K^+ \text{ in filtrate, mg/L} = \frac{1000 (25 - \text{QAS, cm}^3)}{\text{mud filtrate, cm}^3} \quad (a)$$

If correction factor is necessary:

$$K^+ \text{ in filtrate, mg/L} = 1000 \left[ \frac{25 - (\text{CF}) (\text{QAS, cm}^3)}{\text{mud filtrate, cm}^3} \right] \quad (b)$$

$$KCl \text{ in filtrate, lb/bbl} = \left[ \frac{(K^+ \text{ in filtrate, mg/L})}{1500} \right] \quad (c)$$

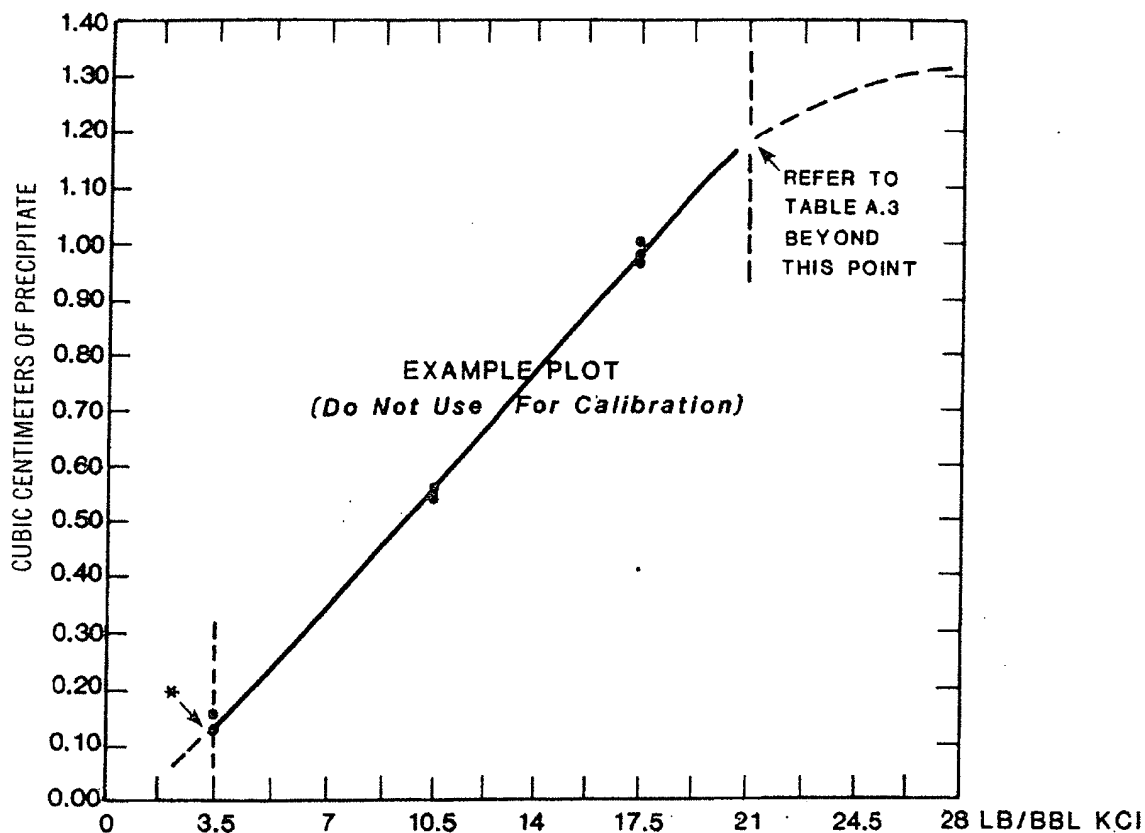


FIG. A.6  
EXAMPLE PLOT  
CALIBRATION CURVE FOR POTASSIUM CHLORIDE

NOTE: Do not use this procedure for concentrations less than 3.5 lb/bbl of KCl.



## APPENDIX B

### SHEAR STRENGTH MEASUREMENT USING SHEAROMETER TUBE

#### B.1 Description

a. Experience has shown that some drilling muds tend to develop excessive shear strength under static conditions, especially at elevated temperatures. Excessive shear strength results in high pump pressures to "break circulation," and may therefore result in loss of circulation. High shear strength may also cause difficulties in logging, perforating, and other "down hole" operations.

b. The following technique can be used to determine the tendency and estimate the extent to which the mud will develop excessive shear strength. This shear strength measurement is normally made on a static heat-aged mud sample. Aging temperatures are therefore selected to be near the estimated bottom-hole temperature of the well. Aging cells or vessels meeting the pressure and temperature requirements for the test are required.

#### B.2 Equipment

##### a. Stainless steel shearometer tube

Length .....	3.5 in. (89 mm)
Outside diameter .....	1.4 in. (36 mm)
Wall thickness .....	0.008 in. (0.2 mm)

*NOTE: A slight outside taper on the bottom of the shear tube has been found to improve reproducibility of the test results.*

##### b. Platform for weights

##### c. Set of gram weights

##### d. Ruler, in

#### B.3 Procedure

a. The shear tube and platform are placed and balanced carefully on the surface of the aged sample cooled to room temperature. It may be necessary to shift the weights on the platform to assure the tube's initial penetration into the mud is vertical. If a crust develops on the heat aged sample, this crust should be gently broken before placing the shear tube in place for test.

b. Sufficient gram weights are placed carefully on the platform to start the downward movement of the shear tube. Unless too much weight is added, the tube will stop its downward travel at the point where the shear strength of the aged mud against the surface of the tube is sufficient to support the applied weight (see Fig. B.1). It is desirable to submerge at least one half the length of the tube.

c. Record the total weight in grams which includes the platform and weights. Measure the portion of the tube submerged in the fluid in inches. The length of the tube submerged can be most accurately determined by measuring the length of the nonsubmerged portion while the tube is at its maximum penetration depth. A small ruler held at the mud surface and alongside the tube will facilitate this measurement. The length of the tube minus the exposed length equals the submerged portion.

#### B.4 Calculation

$$\text{a. Shear strength (S), } = \frac{3.61 (Z + W)}{L} - 0.256 A \quad (\text{a})$$

lb/100 ft<sup>2</sup>

Where: S = shear strength, lb/100 ft<sup>2</sup>  
 Z = weight of shear tube, g  
 W = total shear weight, g  
       (sum of platform and weights)  
 L = submerged length of shear tube, in.  
 A = mud weight, lb/gal

Reference: Watkins, T. E. and Nelson, M. D., "High Temperature Gelation of Drilling Fluids," *AIME Transactions* Vol. 193, 1953, pp. 213-218.

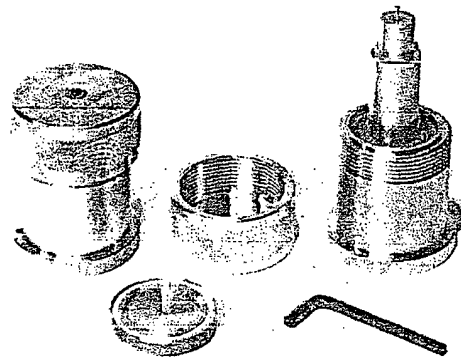


FIG. B.1  
SHEAR STRENGTH APPARATUS



## APPENDIX C RESISTIVITY

**C.1 Description** Control of the resistivity of a mud and mud filtrate may be desirable to better evaluate formation characteristics from electric logs.

### C.2 Equipment

a. Direct reading resistivity meter or similar resistivity meter. Follow manufacturer's instructions for current source, calibration, measurement and calculations (see Fig. C.1 and C.2).

b. Calibrated resistivity cell.

c. Thermometer: 32-220°F (0-105°C)

### C.3 Procedure

a. Fill the clean, dry resistivity cell with freshly stirred mud or mud filtrate.

**CAUTION:** No air or gas should be entrained in sample.

b. Connect cell to meter.

c. Measure resistance in ohm-meters (direct reading) or ohms (not direct reading). Meter or manufacturer's instructions will indicate type of reading.

d. Measure the temperature of sample to nearest degree F (C).

e. Clean the cell. Scrub with brush and detergent if necessary. Rinse with distilled water and allow to dry.

### C.4 Calculation

a. Report the mud resistivity  $R_m$  or filtrate resistivity  $R_{mf}$  in ohm-meters to nearest 0.01 ohm-meter.

b. Report the sample temperature in degrees F (C). If reading is in ohms, convert to ohm-meters by:

$$\text{Resistance} = (R, \text{ohms}) (\text{cell constant}, \text{m}^2/\text{m}) \quad (\text{a})$$

ohm-meter

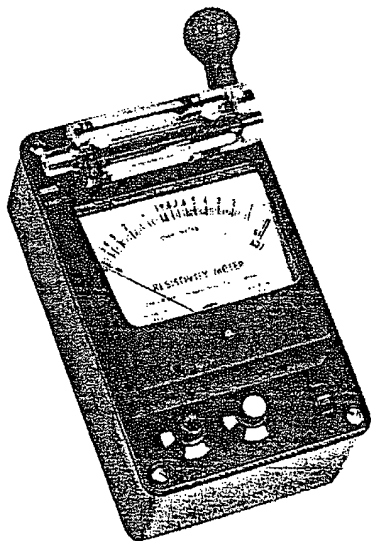


FIG. C.1  
DIRECT-READING RESISTIVITY METER

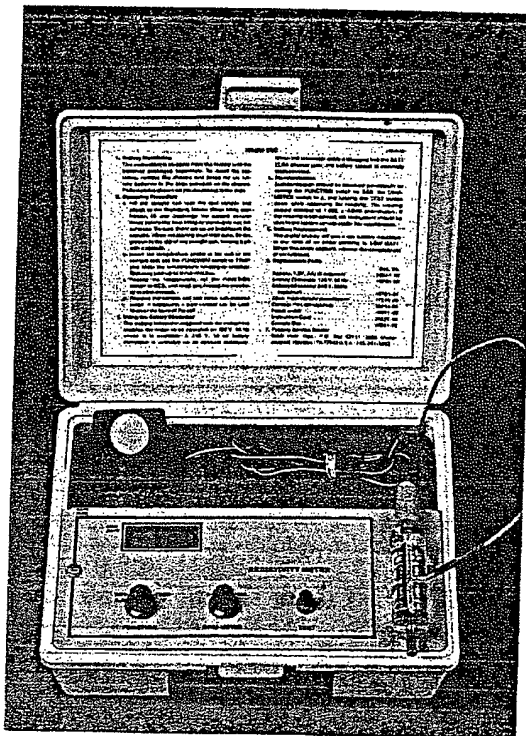


FIG. C.2  
DIRECT-READING RESISTIVITY METER

## APPENDIX D PROCEDURE FOR REMOVAL OF AIR OR GAS FROM MUD PRIOR TO TESTING

**D.1 Description** The majority of drilling muds require no special equipment to remove entrained air or gas prior to testing. Usually, gentle agitation together with a few drops of an appropriate defoamer are all that is necessary. Stirring with a spatula or pouring back and forth is sufficient in most cases. When a mud is encountered that retains air or gas after the preceding steps have been taken, the following procedure can be taken to deaerate the mud.

**NOTE:** If mud density is the only property desired, the Pressurized Fluid Density Balance described in API RP B-1, Par. 1.6 through Par. 1.10 may be used. This unit is illustrated in Fig. 1.3.

### D.2 Equipment

a. A device is necessary which can be evacuated. The equipment shown in Fig. D.1 is available. Follow the manufacturer's operating instructions.

b. Defoamer

### D.3 Procedure

a. Fill clean, dry reservoir about one-half full with the air-cut mud.

b. Add several drops of defoamer to the mud surface.

c. Insert stirrer and cap; cover with gasketed lid.

d. Affix vacuum line from the pump to the instrument and hold about 5 inches-of-mercury vacuum.

e. Increase vacuum to 25-27 inches of mercury and proceed according to the manufacturer's instructions.

f. When mud has been deaerated, partially relieve vacuum to about 15-10 inches-of-mercury vacuum and observe mud for air bubbles.

g. If deaeration is not sufficient, repeat D.3e and D.3f until air is removed.

h. With cylinder on end, relieve vacuum completely and remove mud sample for testing.

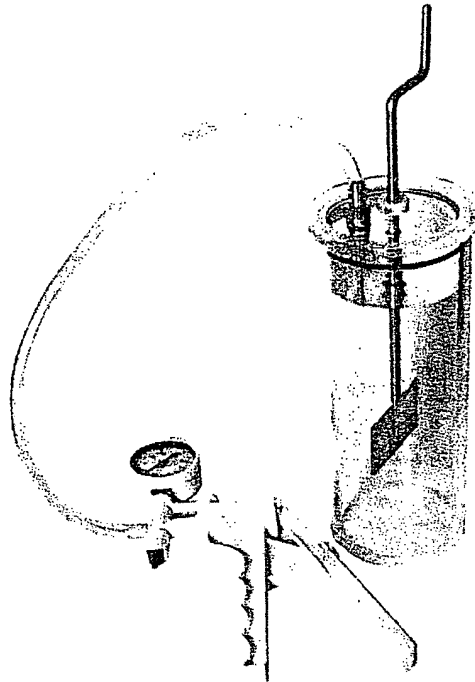


FIG. D.1  
INSTRUMENT FOR AIR OR GAS REMOVAL

## APPENDIX E DRILL PIPE CORROSION RING COUPON

### E.1 Description

a. The placement of corrosion test rings in the drill string is one of the more common techniques used to evaluate the corrosiveness of drilling fluid environments on the drill string and other steel equipment. Removal and examination of these rings after a period of exposure downhole can be highly informative as to the corrosiveness of the drilling fluid as well as to the type of corrosion encountered. An examination of scales and pits on the exposed rings gives clues as to the cause of the corrosion, thus aiding in choosing proper remedial action.

b. The ring technique is specifically designed for detection of the type of corrosion characterized by metal loss whether it be localized pitting or generalized attack. The test ring is not designed to give information relating to hydrogen embrittlement, stress corrosion cracking, or other forms of fracture formation except in the manner in which pitting may relate to these failures.

### E.2 Equipment

a. Ring construction: The ring-type drill string corrosion coupon, or corrosion ring, should be machined to fit in the tool box recess, at the end of the pin, and should have a bore the same as that of the tool joint to minimize turbulence.

b. Ring composition: To avoid galvanic corrosion, the ring should be made from steel identical to that of the tool joint in which it is placed. Such a requirement is impractical and use of a steel that is similar in chemical composition, such as AISI 4130, is recommended.

**NOTE:** If steel other than AISI 4130 is used, it should be identified on the report form.

*The rings are normally cut from tubes that have not been quenched and tempered. The similarity in composition of the 4130 steel and the tool joint should be adequate to minimize galvanic effects and provide useful data.*

c. Ring marking: The rings should be stenciled with a serial number for permanent identification.

d. Ring preparation: The rings should be scrubbed with a stiff fiber bristle brush and detergent solution, rinsed with clean water and with anhydrous acetone or methanol. Allow to dry, weigh to nearest milligram, and record this weight on the report form. Store the ring in a dry container, such as a desiccator, to prevent corrosion. The corrosion rings should be shipped to the field in sealed envelopes or wrappers to minimize atmospheric corrosion.

### E.3 Procedure

a. Drill pipe corrosion rings should be kept in the drill string for a minimum of 40 hours. A normal time for exposure is 100 hours. Exposure periods of less than

40 hours should not be used because initial corrosion rates may be unusually high and can give misleading data. The ring is usually placed in the tool joint at the top of the first stand above the drill collars and can be left in the drill string for more than one bit run. An additional ring can be placed in the kelly saver sub to monitor corrosion at that point. Care should be taken to ensure that the box recess is clean to prevent interference with proper make-up of the joint and to avoid damage to the ring. In some instances specially manufactured subs have been used for the ring placement in the string. During installation the ring should be handled with clean, dry gloves.

b. The drill pipe corrosion coupon form should be filled out completely. Each form should have a space for ring material, drilling fluid properties, type of corrosion, location of ring in the drill string, initial weight, time, depth in, depth out, ring number, color of scale, and any other information of significance in the specific test. The form may be printed on a mailing envelope for the ring or on a separate form to be enclosed with the ring.

c. The drilling fluid residue should be removed from the coupon by wiping with a cloth when the ring is pulled from the drill string. The ring should be examined for severity of corrosion or mechanical damage. If severe corrosion is evident, the cause of the corrosion should be determined promptly so remedial action can be taken. Following visual observation, immediately coat the coupon with oil or other available grease. (Do not use thread compound for this purpose.) Place in a plastic bag and then in the mailing envelope.

d. Before proceeding with a quantitative evaluation of corrosion of the ring, the ring should be rinsed with suitable solvent, such as acetone or petroleum ether, to remove the oil applied to the ring on location. Prior to cleaning for weighing, a spot test should be made for corrosion by-products and mineral scale. For example, the surface can be examined qualitatively for sulfides by the acid arsenite test (see Corrosion of Oil- and Gas-Well Equipment, API, Dallas (1958)). The rings should be cleaned with a detergent solution and a stiff fiber bristle brush. It may be necessary to dip the ring for 5-10 seconds in inhibited 10-15% hydrochloric acid one or more times to remove corrosion products. The ring should be scrubbed with detergent solution after each acid dip. Rinse thoroughly with clean water and then with anhydrous acetone or methanol. Allow to dry prior to weighing. Very abrasive materials or strong, uninhibited acids should not be used. An ultrasonic bath can be useful in cleaning the rings.

e. After the preweighed drill pipe corrosion coupon has been properly cleaned and the corrosion film and type of attack noted, the ring should be reweighed to nearest milligram and the weight loss determined. If

significant loss of metal due to mechanical damage is evident, it should be noted and taken into consideration in evaluation of the ring. The corrosion rate may be reported as kilograms per square meter per year, pounds per square foot per year, or mils per year. Formulas for calculating for corrosion rate are given in Par. E.4.

#### E.4 Comments on Visual Examination

a. If visual corrosion is evident, it will normally be detectable as pitting corrosion. Uniform attack or general corrosion can best be determined by a weight loss measurement. Mechanical damage to the ring will most often be evidenced by cuts or dents on the outer surfaces of the ring. In some cases, the ring will exhibit a series of dents and worn spots, indicating considerable movement of the ring in the box recess.

b. In assessing the magnitude of the corrosion rates as calculated from weight loss measurements, it should be remembered that the rate is also influenced by the erosive effects of the drilling fluid. Since the bore of the ring is exposed to the mud pumped down the drill pipe, the loss of metal includes that removed by erosion as well as from corrosion. Loss from erosion can be substantial when the drilling fluid contains a high concentration of sand.

c. Examination of the ring may reveal a few deep pits with a relatively low weight loss. This condition would indicate a rather severe corrosion problem even though the calculated corrosion rate would be considered low.

#### E.5 Calculation

$$\begin{aligned} \text{a. kg/m}^2/\text{yr} &= \left[ \frac{\text{wt. loss, mg}}{1,000,000} \right] \left[ \frac{10,000}{\text{area, cm}^2} \right] \left[ \frac{365}{\text{days exposure}} \right] \quad (\text{a}) \\ &= \frac{87.60 (\text{wt. loss, mg})}{\text{area, cm}^2 (\text{hours exposed}^{**})} \quad (\text{b}) \end{aligned}$$

$$= \frac{13.58 (\text{wt. loss, mg})}{\text{area, in}^2 (\text{hours exposed}^{**})} \quad (\text{c})$$

$$\text{b. lb/ft}^2/\text{yr} = \left[ \frac{\text{wt. loss, mg}}{453,600} \right] \left[ \frac{144}{\text{area, in}^2} \right] \left[ \frac{365}{\text{days exposure}} \right] \quad (\text{d})$$

$$= \frac{2.781 (\text{wt. loss, mg})}{\text{area, in}^2 (\text{hours exposed}^{**})} \quad (\text{e})$$

$$\text{c. mils/yr} = \frac{\text{wt. loss, mg}}{\left[ \frac{16,387}{\text{cm}^3/\text{in}^3} \right] \left[ \frac{(\text{specific gravity})}{\text{g/cm}^3} \right] \left[ \frac{(\text{area})}{\text{in}^2} \right] \left[ \frac{(\text{year, days/365})}{\text{days}} \right]} \quad (\text{f})$$

For steel coupons with a specific gravity of 7.86, the formula can be reduced to:

$$\text{mils/yr} = \frac{68.33 (\text{wt. loss, mg})}{\text{area, in}^2 (\text{hours exposed}^{**})} \quad (\text{g})$$

\*Total surface area of the ring is used in these calculations.

\*\*Time used based on total time in the drill string.

d. Following are the conversion rates between the various units for steel coupons (specific gravity 7.86):

$$\begin{aligned} \text{mils/yr} &= 24.62 (\text{lb/ft}^2/\text{yr}) & (\text{h}) \\ \text{mils/yr} &= 5.03 (\text{kg/m}^2/\text{yr}) & (\text{i}) \\ \text{lb/ft}^2/\text{yr} &= 0.04 (\text{mils/yr}) & (\text{j}) \\ \text{lb/ft}^2/\text{yr} &= 0.20 (\text{kg/m}^2/\text{yr}) & (\text{k}) \\ \text{kg/m}^2/\text{yr} &= 0.20 (\text{mils/yr}) & (\text{l}) \\ \text{kg/m}^2/\text{yr} &= 4.90 (\text{lb/ft}^2/\text{yr}) & (\text{m}) \end{aligned}$$

e. Corrosion rings available from drilling fluid service companies or corrosion test laboratories are generally supplied with a multiplication factor which includes the metal density and surface area of the ring. Thus, the weight loss divided by the exposure time need only be multiplied by the supplied factor to obtain the corrosion rate.

## APPENDIX F SAMPLING, INSPECTION, AND REJECTION

**F.1 Description.** The following procedure is the official sampling, inspection, and rejection method for materials conforming to API specification. It is applicable to barite, hematite, bentonite, nontreated bentonite, attapulgite, sepiolite, technical-grade low viscosity carboxymethylcellulose, technical-grade high viscosity carboxymethylcellulose, and OCMA grade bentonite and is to be used in the event of product property dispute.

### **F.2 Procedure — Powdered Material In Packages**

a. The number of samples taken to be combined for the test sample (see Par. F.4) shall be 15 samples of one pound (0.45 kg) or more per lot.

b. For each lot of 1,000 packages or less, 15 packages shall be sampled.

c. The sampling may be carried out by either of the following methods as agreed upon by contracting parties.

1. A sample weighing at least 1 lb (0.45 kg) shall be taken from the top of each package.
2. A sampling tube, capable of taking a core not less than 1 in. (2.5 cm) in diameter shall be used. The tube shall be inserted into the package being sampled so that it will take a core of material for substantially the entire length of the package.

### **F.3 Procedure — Powdered Material in Bulk**

a. In the collection of samples from cars or storage bins containing 25 to 100 tons (22,700 to 90,900 kg), a sampling tube capable of taking a core not less than 1 in. (2.5 cm) in diameter shall be used. The tube shall be of sufficient length to permit taking a sample substantially from the top to the bottom of the mass being sampled.

b. Fifteen samples shall be taken from each lot (each car or bin shall be considered one lot). If the dimensions of the container are such that the sample cannot be taken in this manner, the sample shall be taken by a method agreed upon by the contracting parties.

c. From bins containing less than 25 tons (22,700 kg), at least one sample shall be taken from each container up to lots of 10 containers, and the total number of samples taken from each lot of 100 tons (90,900 kg) or less shall be not less than 10.

### **F.4 Procedure — Test Sample**

a. The samples from each lot shall be combined, mixed, and quartered or riffled to furnish a test sample of 15 lb (6.8 kg) which shall be divided into 3 equal portions.

b. Each portion shall be sealed in a suitable airtight moisture-proof container. One sample shall be delivered to the purchaser, one to the supplier, and the third sample shall be retained for a reference test, if required.

### **F.5 Procedure — Test Results**

a. Each party shall make one measurement on his test sample.

b. A control or known reference standard, such as the API Test Calibration Barite, shall be tested in the same manner, at the same time as part of the test series. If this test result is within the acceptable range, the test results can be considered valid and reported. If it is not within the acceptable range, the results are to be considered suspect, and the cause found and corrected before retesting.

**F.6 Procedure — Inspection.** The purchaser's inspector shall be afforded, without charge, all reasonable facilities for careful sampling and inspection. A period of six days shall be allowed for sampling and completion of acceptance test.

**F.7 Procedure — Rejection.** Rejection of material, based on failure to pass the test prescribed in the specifications, shall be reported to the supplier immediately upon completion of the tests and the cause for rejection shall be stated.

### **F.8 Procedure — Retesting**

a. Either of the contracting parties may make claim for retest within one week of the date of the original test report. The expense of the retest shall be borne by the party demanding such retest.

b. Should the contracting parties be unable to reach agreement, the third sample of material shall be delivered unopened, to a mutually satisfactory referee laboratory for tests, and the results of this referee shall be binding on both parties.

## APPENDIX G RIG SITE SAMPLING

### G.1 Description

a. The following is a recommended procedure for obtaining test samples at the rig site from specific tank-truck loads. It is not the official procedure approved for sampling materials for API specification testing.

b. Upon agreement by contracting parties this procedure can be used for testing the properties of material as delivered to the rig-site. It has been used on barite and weight material with good results and should be equally applicable to similar bulk shipments of bentonite, attapulgite, and sepiolite.

### G.2 Equipment — Bottom Flow Sampling (Preferred)

a. Side-stream sampler: shown in Fig. G.1

b. Canvas bag: 5½ in. (140 mm) diameter and 12 in. (300 mm) long

c. Container: tie-bag or other suitable container, so constructed as to retain all fines.

### G.3 Procedure — Bottom Flow Sampling (Preferred)

a. Connect a side-stream sampler to the truck outlet.

b. Take three 3-5 lb (1.4-2.3 kg) samples while unloading, spaced so as to be taken when approximately ¼, ½, and ¾ of the load has been transferred.

NOTE: One 9-15 lb (4.1-6.8 kg) sample can be taken with the recognition that there is a chance of obtaining a nonrepresentative sample. The greatest potential for error in sampling is contamination from prior shipments of material in the same vessel. This potential error can be minimized by following the instructions closely. Do not take samples from the very top surface or from the very first material out of the bottom. A second potential for error in sampling is taking material from the side-stream sampler when the transfer hose is only partially full. This can occur when the truck is nearly empty or material flow has been interrupted. Segregation has been shown to occur in these instances.

c. Before each individual sample is taken, open the valve momentarily to flush out any material trapped in the side pipe. Tie a clean canvas bag to the sampler and fill it with one valve opening operation.

NOTE: Partial closing of the air-jet valve on the bottom outlet prior to taking the sample often helps to get a full sack with a minimum of dusting.

d. Carefully transfer the sample from the canvas bag to a tie-bag or other suitable container. Label this container with all pertinent information.

e. Empty the canvas bag before taking the next sample. Always use a clean or new canvas bag for each truck.

### G.4 Equipment — Grab Sampling

a. Sample scoop: shown in Fig. G.2

1. 4-6 in. (100-150 mm) deep
2. 12-18 in. (300-450 mm) long

b. Container: tie-bag or other suitable container, so constructed as to retain all fines.

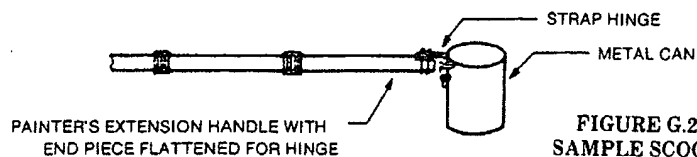
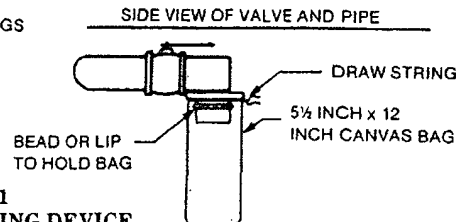
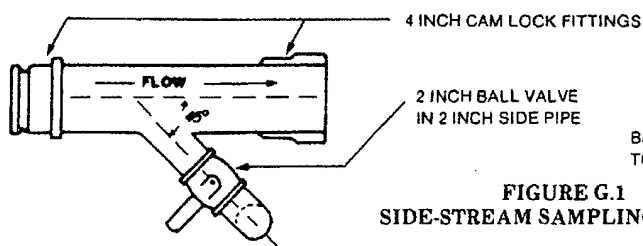
### G.5 Procedure — Grab Sampling

a. Open the top hatch carefully. Do not let the cover bump the top of the tank.

b. Remove the top surface material under the hatch to form a trench 1½ times wider than the sample scoop.

c. Scoop out a 3-5 lb (1.4-2.3 kg) sample from the bottom of this trench and transfer to a tie-bag or other suitable container. Label the container.

d. Repeat this operation at two other spots on the top of the material near the mid point between the middle and the ends of the truck tank.



# APPENDIX H METRIC "SI" UNIT CONVERSION TABLE

PROPERTY	TRADITIONAL UNIT	RECOMMENDED SI UNIT	SYMBOL	CONV. FACTOR	EXAMPLE
Mechanical Properties					
Depth	feet	metre	m	0.3048	10,000 ft = 3048 m
Hole Diameter	inch	millimetre	mm	25.4	12 1/4 in. = 311 mm
Pipe Diameter	inch	millimetre	mm	25.4	4 1/2 in. = 114 mm
Bit Size	inch	millimetre	mm	25.4	12 1/4 in. = 311 mm
Weight on Bit	pound	newton	N	4.4	20,000 lb = 88,000 N
Rotary Table Speed	rpm	*revolutions per minute	r/min	1	45 rpm = 45 r/min
Nozzle Size	1/32 inch	millimetre	mm	0.79	10/32 in. = 7.9 mm
Nozzle Velocity	feet/sec	metre/sec	m/s	0.3048	400 ft/sec = 122 m/s
Drilling Rate	feet/hour	*metre/hour	m/h	0.3048	30 ft/h = 9 m/h
Volume	barrels	cubic metre	m <sup>3</sup>	0.159	3000 bbl = 477 m <sup>3</sup>
Liner Size	inch	millimetre	mm	25.4	6 1/2 in. = 165 mm
Rod Diameter	inch	millimetre	mm	25.4	2 1/4 in. = 57 mm
Stroke length	inch	millimetre	mm	25.4	16 in. = 406 mm
Pump Output	barrel/minute	*cubic metre/min	m <sup>3</sup> /min	0.159	8.5 bbl/min = 1.35 m <sup>3</sup> /min
	gallon/minute	*cubic metre/min	m <sup>3</sup> /min	.00378	357 gpm = 1.35 m <sup>3</sup> /min
Pump Pressure	pound/sq inch	kilopascal	kPa	6.9	2500 psi = 17,300 kPa
Annular Velocity	feet/min	*metre/minute	m/min	0.3048	200 ft/min = 61 m/min
Slip Velocity	feet/min	*metre/minute	m/min	0.3048	20 ft/min = 6.1 m/min
Temperature	degree Fahrenheit	degree Celsius	°C	(°F-32)/1.8	80°F = 27°C
Funnel Viscosity					
	degree centigrade	degree Celsius	°C	1.0	
	seconds/quarter	seconds/litre	s/L	Units cannot normally be converted	
	seconds/1000 cm <sup>3</sup>	seconds/litre	s/L	1.0	
Mud Weight					
	pounds/gallon	kilogram/cubic metre	kg/m <sup>3</sup>	120	10 lb/gal = 1200 kg/m <sup>3</sup>
	pounds/ft <sup>3</sup>	kilogram/cubic metre	kg/m <sup>3</sup>	16	74.8 lb/ft <sup>3</sup> = 1200 kg/m <sup>3</sup>
Pressure Gradient	psi/ft	kilopascal/metre	kPa/m	22.6	0.52 psi/ft = 11.8 kPa/m
Hydrostatic Head	psi	kilopascal	kPa	6.9	4000 psi = 27,600 kPa
Shear Stress	lb/100 ft <sup>2</sup>	pascal	Pa	0.48	20 lb/100 ft <sup>2</sup> = 960 Pa
	dynes/cm <sup>2</sup>	pascal	Pa	0.100	10 dynes/cm <sup>2</sup> = 1.0 Pa
Shear Rate	reciprocal second	reciprocal second	s <sup>-1</sup>	1.0	
Apparent, Plastic & Effective Viscosity	centipoise	*centipoise	cP		
Yield Point	lb/100 ft <sup>2</sup>	pascal	Pa	0.48	15 lb/100 ft <sup>2</sup> = 7.2 Pa
Gel Strength	lb/100 ft <sup>2</sup>	pascal	Pa	0.48	3 lb/100 ft <sup>2</sup> = 1.44 Pa
Dial Reading	lb/100 ft <sup>2</sup>	pascal	Pa	0.51	dial reading - 10 = 5.1 Pa
Standard V-G Meter	1.065				
Power Law Constants					
	n	dimensionless			
	K	dynes s <sup>n</sup> /cm <sup>2</sup>	mPa-s <sup>n</sup> /cm <sup>2</sup>	100	10 dynes s <sup>n</sup> /cm <sup>2</sup> = 100 mPa-s <sup>n</sup> /cm <sup>2</sup>
		lb/ft s <sup>n</sup> /100 ft <sup>2</sup>	mPa-s <sup>n</sup> /cm <sup>2</sup>	479	1.2 lb s <sup>n</sup> /100 ft <sup>2</sup> = 575 mPa-s <sup>n</sup> /cm <sup>2</sup>
API Filtrate					
	cubic centimetre/30 min	*cubic centimetre/30 min	cm <sup>3</sup> /30 min		
Filter Cake	1/32 inch	millimetre	mm	0.8	3/32 in. = 2.4 mm
Sand Content)	Volume percent	cubic metre/cubic metre	m <sup>3</sup> /m <sup>3</sup>	0.01	10% = 0.1 m <sup>3</sup> /m <sup>3</sup>
Solids Content)					
Oil Content)					
Water Content)					
Particle Size	micron	micrometre	um	1.0	
Chemical Properties					
Ionic Concentration					
	grains/gallon	*milligram/litre	mg/L	17.1	500 grains/gal = 8600 mg/L
	parts/million	*milligram/litre	mg/L	x specific gravity	100,000 ppm of NaCl x 1.0707 = 107,070 mg/L
Alkalinity					
	cubic centimetre/	cubic centimetre/	cm <sup>3</sup> /cm <sup>3</sup>		
P <sub>T</sub> , M <sub>T</sub> , P <sub>1</sub> , P <sub>2</sub>	cubic centimetre	cubic centimetre			
MBT					
	cubic centimetre/	cubic centimetre/	cm <sup>3</sup> /cm <sup>3</sup>		
Methylene Blue Content	cubic centimetre	cubic centimetre			
Miscellaneous					
Additive Concentration	pound/barrel	kilogram/cubic metre	kg/m <sup>3</sup>	2.85	10 lb/bbl = 28.5 kg/m <sup>3</sup>
Corrosion Rate	lb/ft <sup>3</sup> /yr	kilogram/square metre/year	kg/m <sup>2</sup> /yr	4.9	0.9 lb/ft <sup>3</sup> /year = 4.3 kg/m <sup>2</sup> /year
Bentonite Yield	mils/year	millimetre/year	mm/a	.0254	200 mils/year = 5.08 mm/a
Hydraulic HP	barrels/US ton	*cubic metre/metric tonne	m <sup>3</sup> /t	0.175	100 bbl/ton = 17.5 m <sup>3</sup> /t
Screen Size	horsepower	kilowatt	kw	0.746	600 hhp = 450 kw
Mesh	openings/inch	openings/centimetre	openings/cm	0.254	100 mesh = 25.4 openings/cm
Aperture	micron	micrometre	um	1	
Open Area	area percent	square metre/square metre	m <sup>2</sup> /m <sup>2</sup>	0.01	30% = 0.3 m <sup>2</sup> /m <sup>2</sup>
Weight of Drill Pipe	pound/foot	kilogram/metre	kg/m	1.49	19.5 lb/ft = 29.1 kg/m
Oil Muds					
Oil Content)	volume percent	cubic metre/cubic metre	m <sup>3</sup> /m <sup>3</sup>	0.01	10% = 0.1 m <sup>3</sup> /m <sup>3</sup>
Water Content)					
Emulsion Stability	volts	volts	V		
Activity	Dimensionless				
Salinity	ppm	*milligrams/litre	mg/L	specific gravity	250,000 ppm of CaCl <sub>2</sub> x 1.24 = 310,000 mg/L
Aniline Point	degree Fahrenheit	degree Celsius	°C	(°F-32)/1.8	150°F = 66°C

\* Denotes acceptable deviation from API 2564 preferred unit

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